METALLURGIA

THE BRITISH JOURNAL OF METALS

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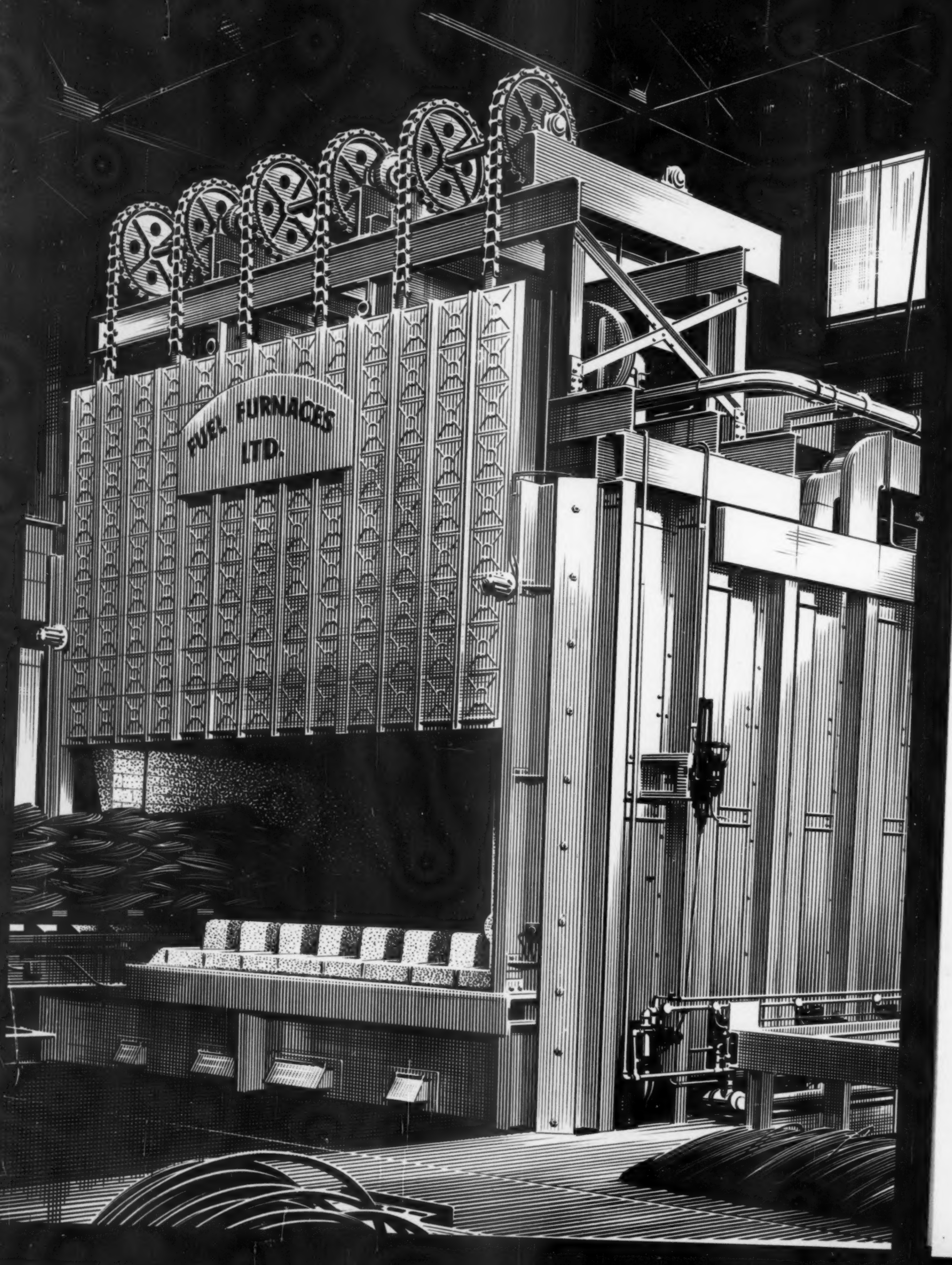
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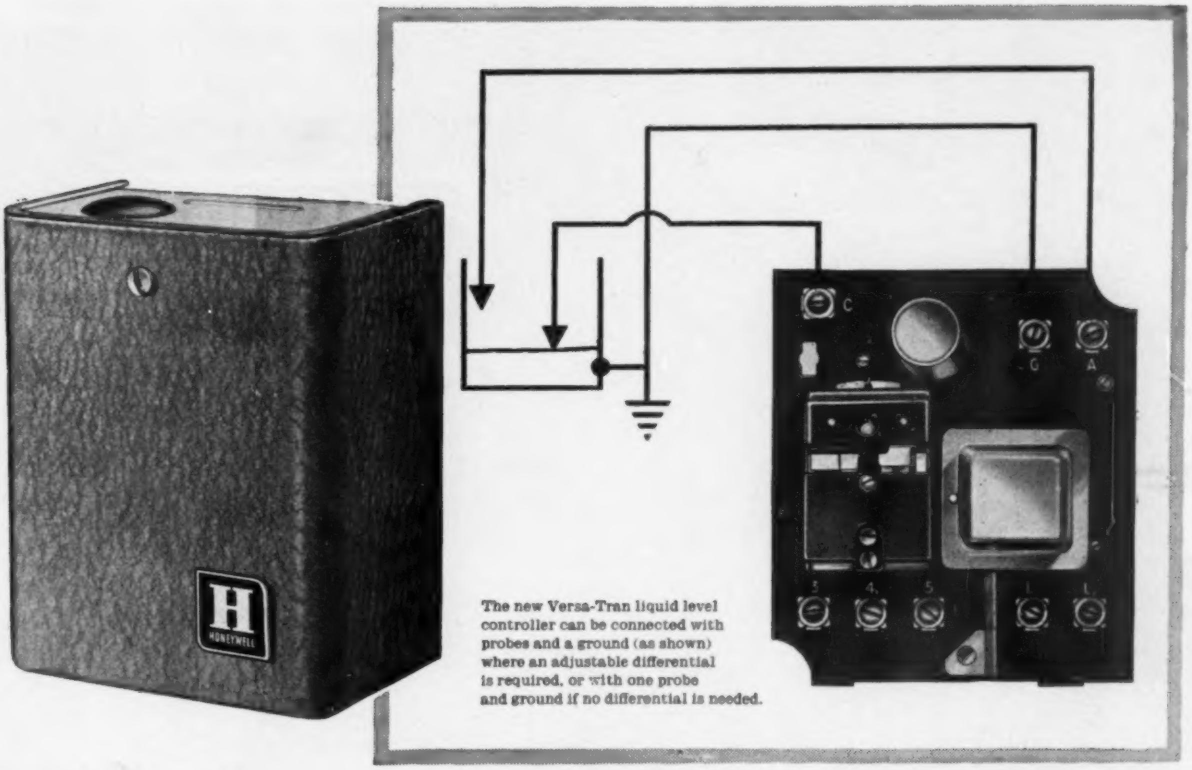

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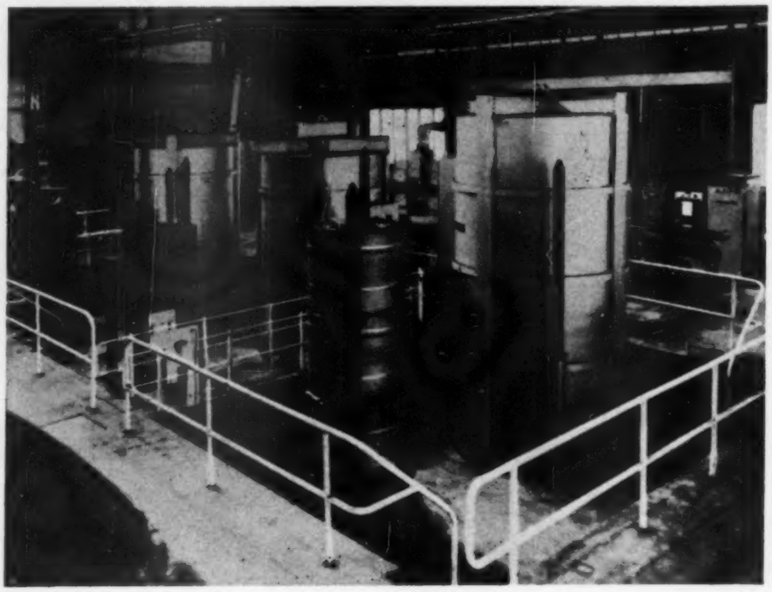

The only company in Great Britain specialising in the production of stainless steel coiled strip, Shepcote Lane Rolling Mills of Sheffield have to be confident that their heat-treatment is not only rigidly controlled but that a continuous seven-day-a-week output is maintained without furnace failure.

As production and the range of products have increased much new plant has been installed, among which are four G.W.B. electrically heated bell furnaces. Hot rolling to a thickness of approximately .128″ from slabs of stainless steel weighing between 2 and 3 tons produces coils up to 54″ diameter and widths ranging from 24½″ to 40½″, and it is after hot rolling that softening in G.W.B. bell furnaces is carried out in order to remove residual hot rolling stresses prior to descaling and cold rolling to lighter gauges.

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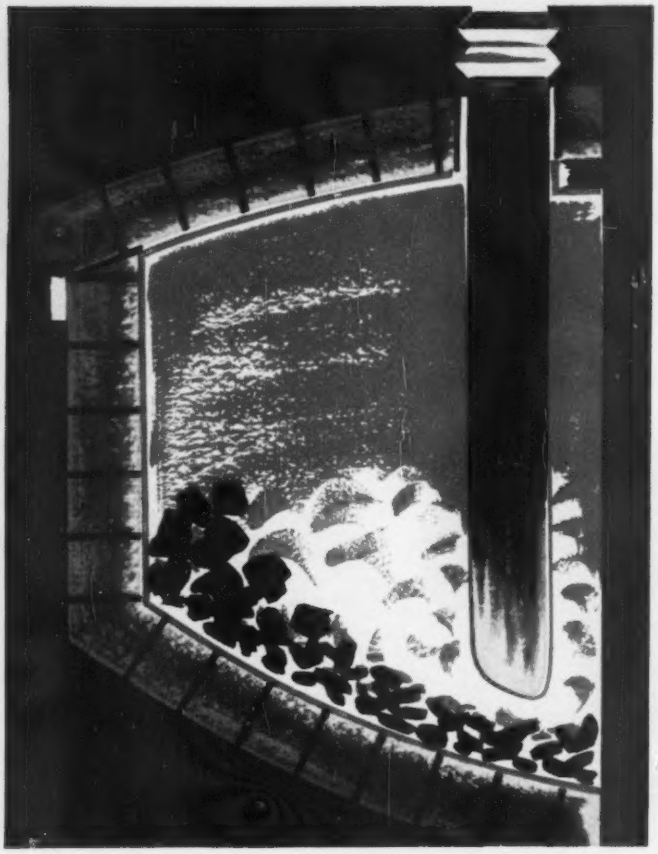


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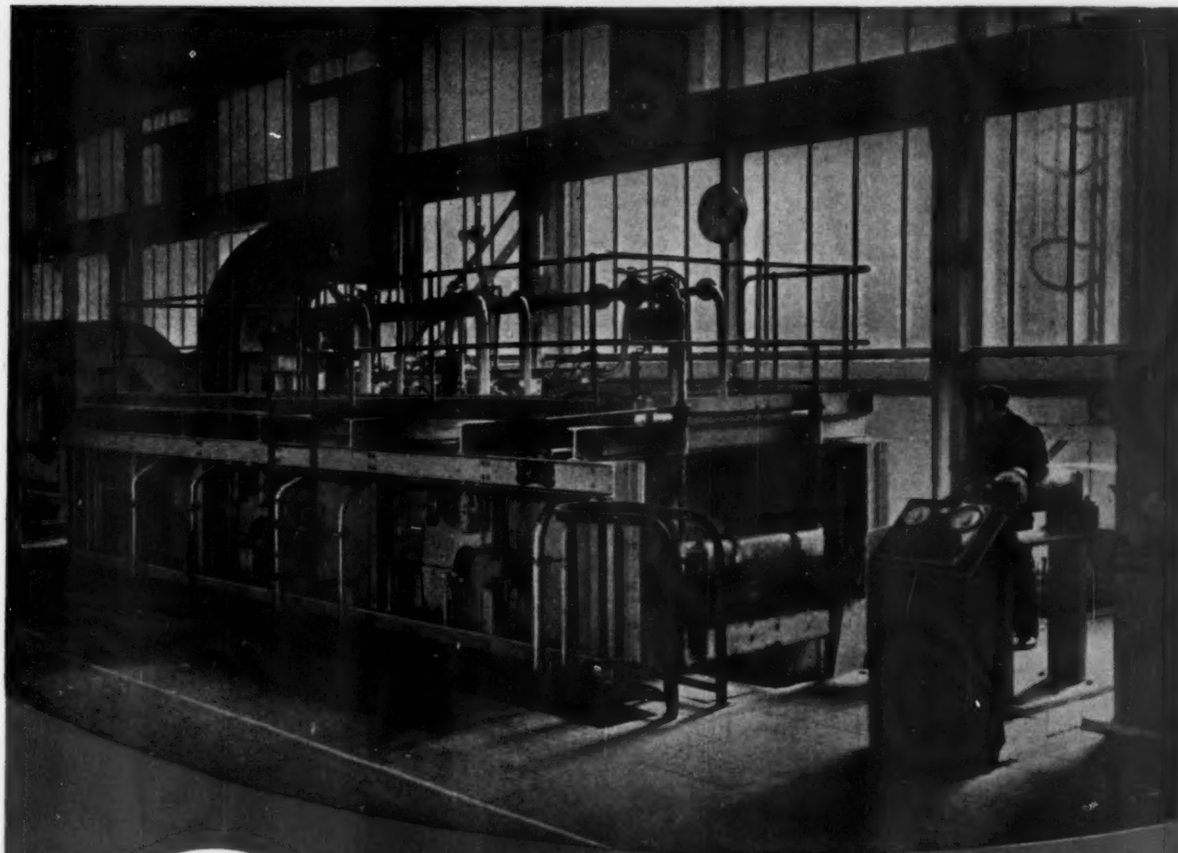


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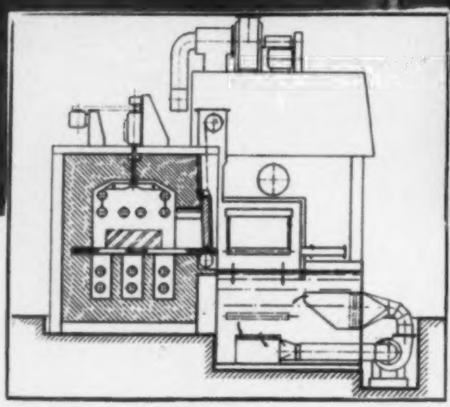

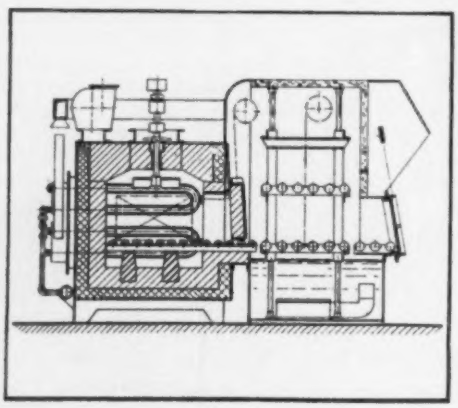

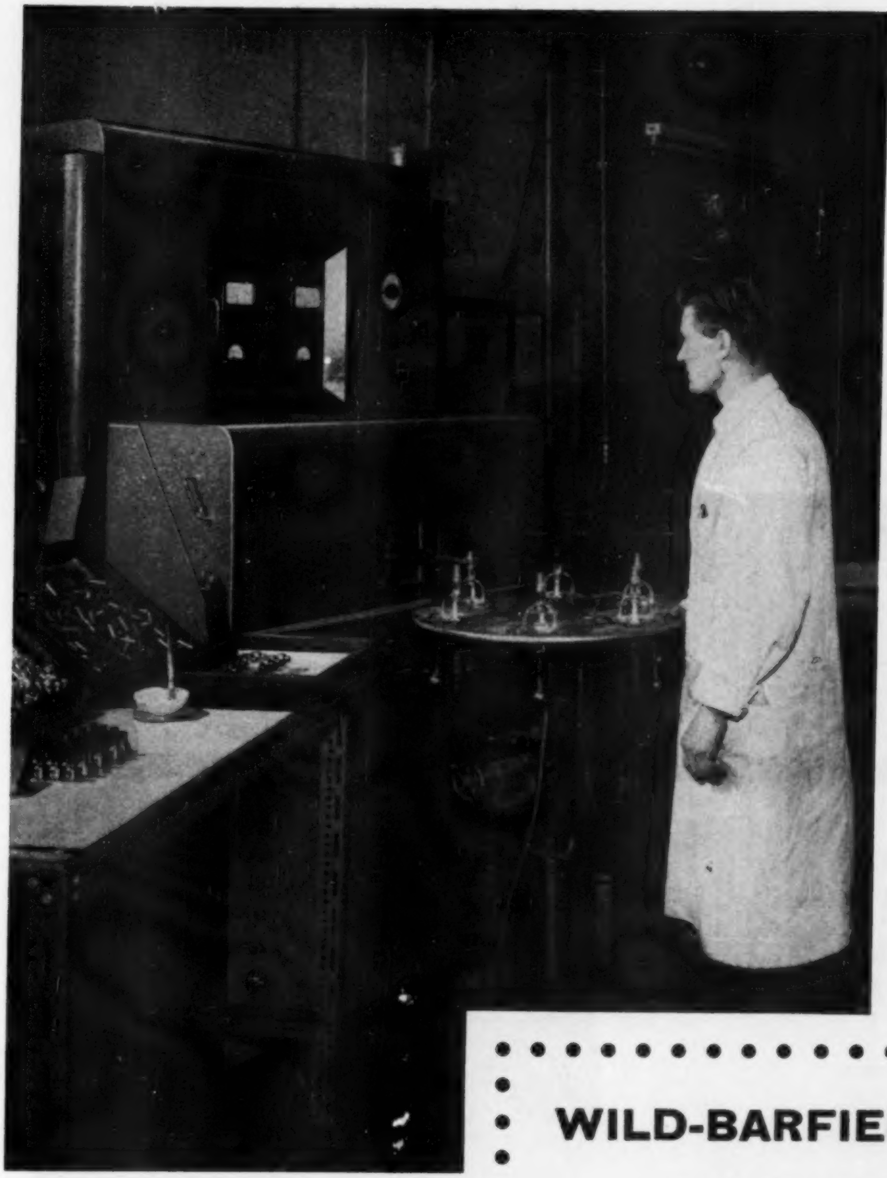

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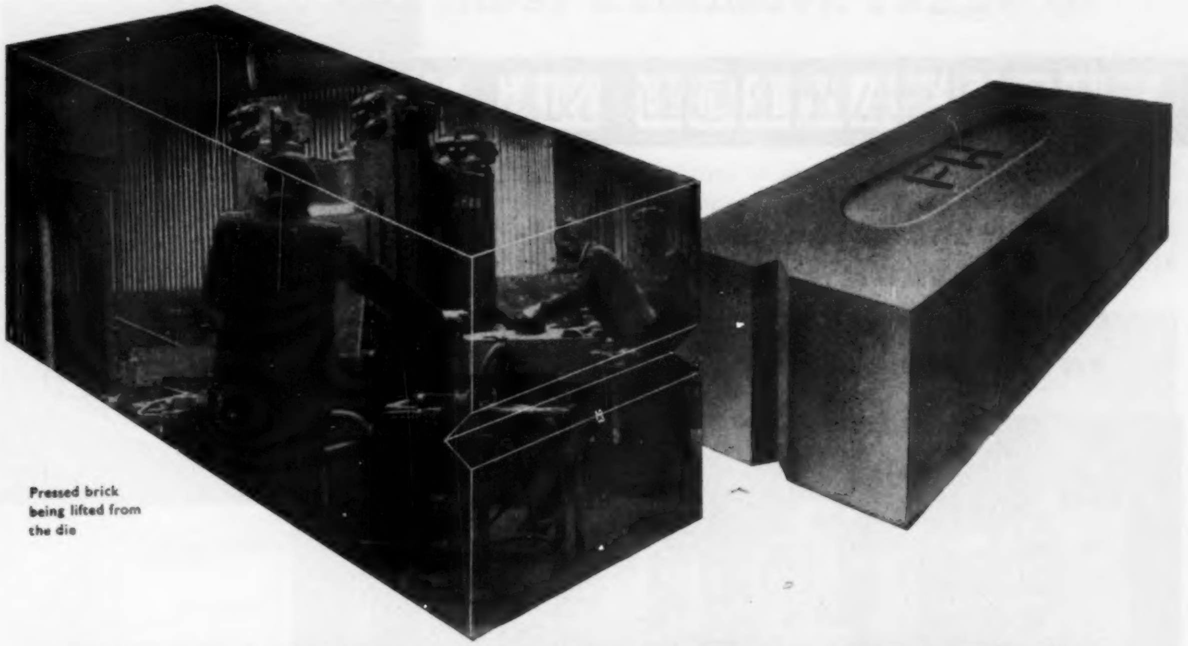

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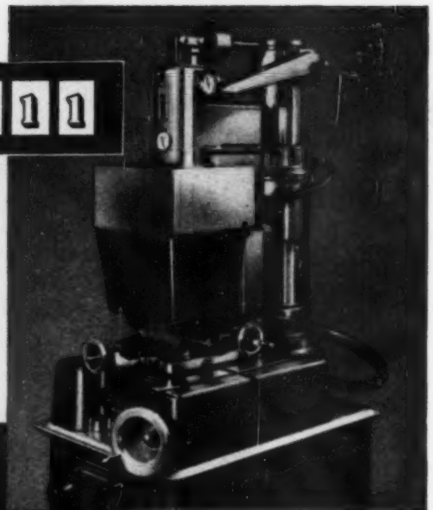

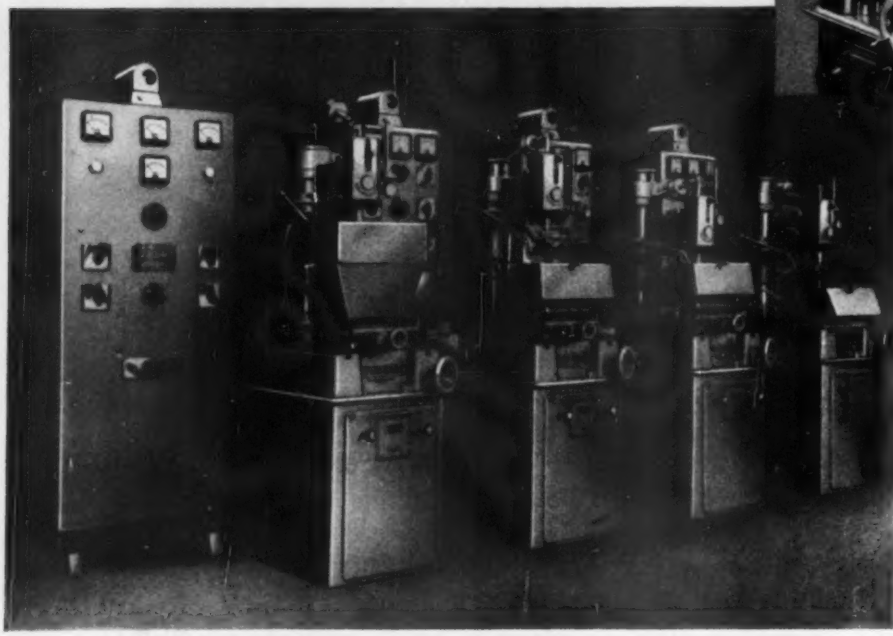

Up to four spark-heads can be operated from one control cabinet. The current is automatically cut off when the electrode has penetrated the workpiece to a predetermined depth allowing one operator to supervise a number of heads.

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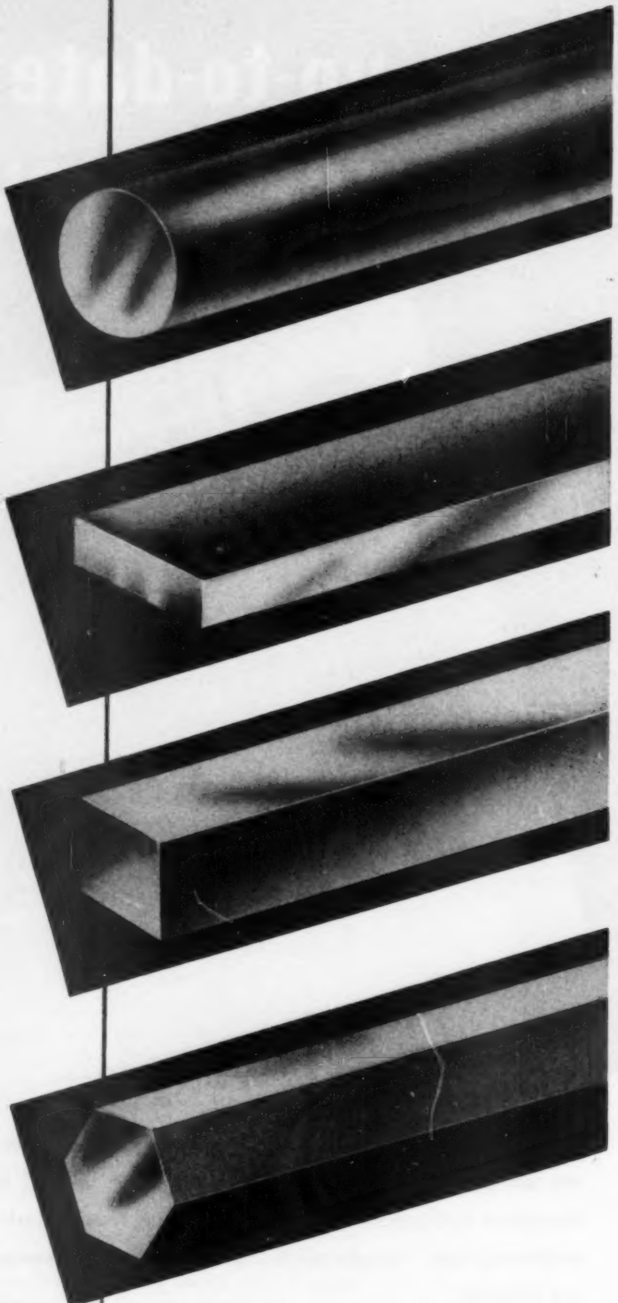

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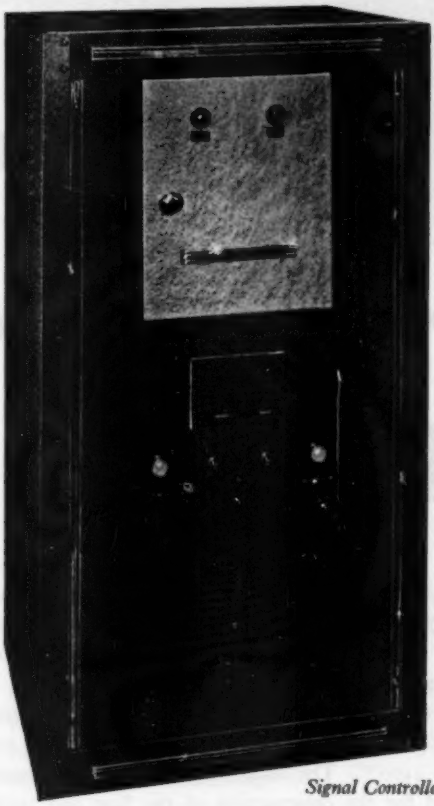

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Autocarb systems automatically adjust the flow of air or enriching hydrocarbon to the furnace or generator in direct response to the dew point of the atmosphere. Specified conditions are automatically maintained. Autocarb systems are simple compact units which indicate, record and control the dew point of gas atmospheres in continuous or batch furnaces and atmosphere generators. They can control dew points to within plus or minus two degrees. On continuous furnaces separate zones of atmosphere control can be established and automatically maintained. If manual control is preferred, the Autocarb line includes simple recorders or indicators. On batch furnaces the Autocarb system will maintain the desired carbon potential for each temperature established in the heating chamber. On endothermic gas generators Autocarb systems automatically and continuously compensate for fluctuations in the composition of the reaction fuel gas supply and the humidity of the reaction air. This keeps constant the dew point of the product gas.

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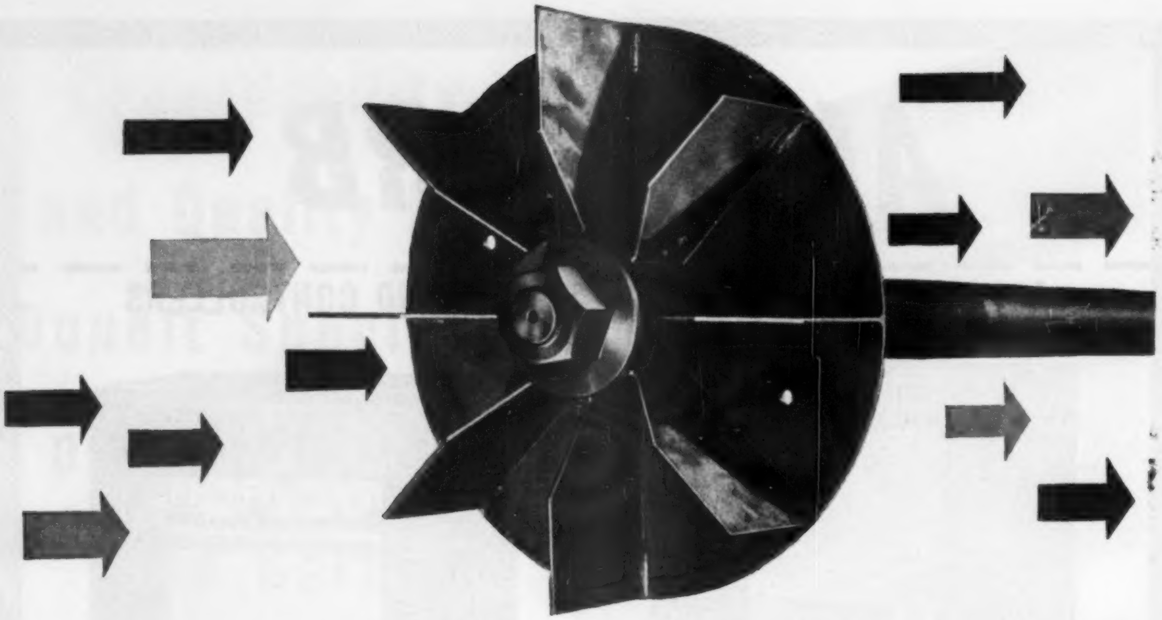

For furnace fans operating at 1100° C

-Nimonic* 75

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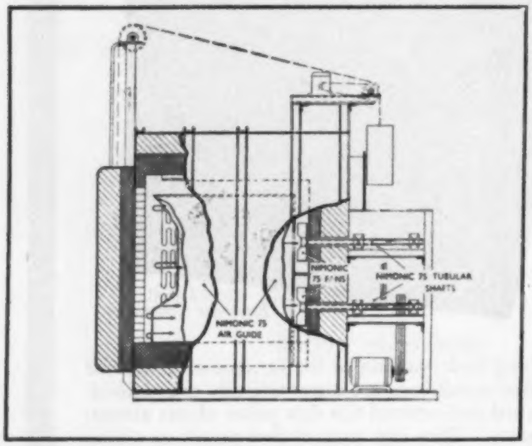

A high-temperature electric furnace of advanced design has been developed by Hedin Limited, for the heat-treatment of components during the overhaul of Rolls-Royce 'Avon' and 'Conway' turbo-jet engines.

To ensure even heat distribution within the furnace, four centrifugal fans are required to operate continuously at temperatures up to 1100°C. The nickel-chromium alloy, Nimonic 75, was selected for the construction of the fans and their drive shafts. The excellent high-temperature properties of Nimonic 75 enable it to resist oxidation and withstand the exacting centrifugal stresses at extreme heat.

TOP
In addition to the forced-circulation fans and drive shafts, Nimonic 75 is used for the air-guide furnace liner which directs circulating air over the heating elements.

INSET
Diagram shows forced-circulation system and the Nimonic 75 hollow fan-shafts cooled by compressed air.

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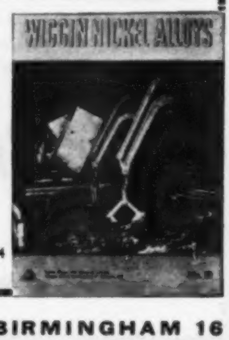

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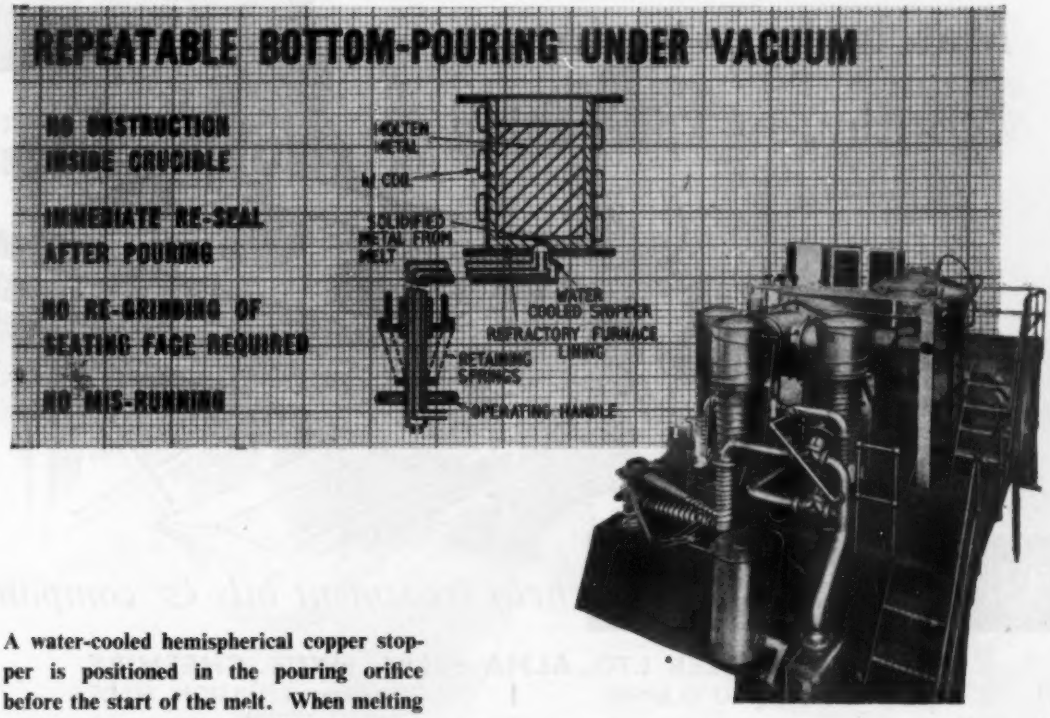
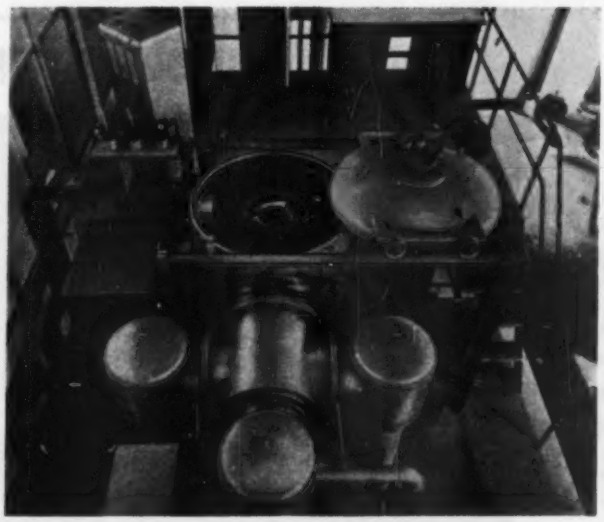


A water-cooled hemispherical copper stopper is positioned in the pouring orifice before the start of the melt. When melting begins, liquid metal is solidified immediately on contact with the stopper; the resulting plug of metal being kept solid by the water cooling supply to the stopper. Before pouring, the stopper is swung clear and the plug melted by conduction from the melt. The stopper can be replaced immediately after pouring since the sealing face remains clean and there are no mating faces to require regrinding. The crucible can thus be recharged quickly and safely without fear of misrunning.

This unique bottom-pouring technique was used on a 200 kg uranium melting furnace constructed by us for the Australian Atomic Energy Commission (illustrated above).



(Right): Photograph taken from above the furnace chamber showing lid removed and the crucible assembly.

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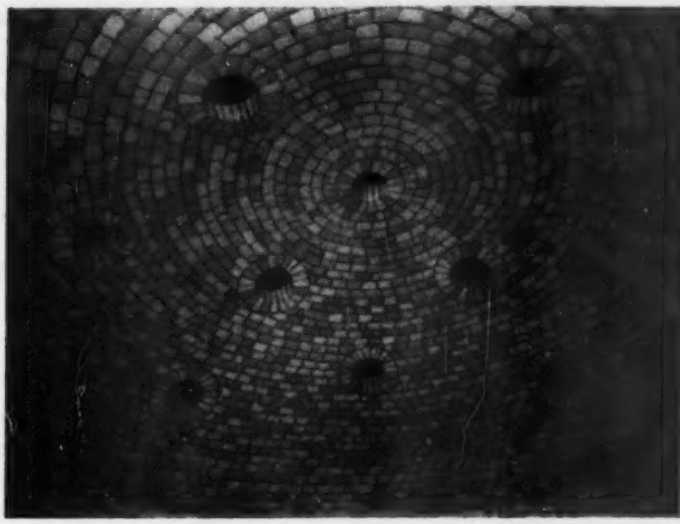

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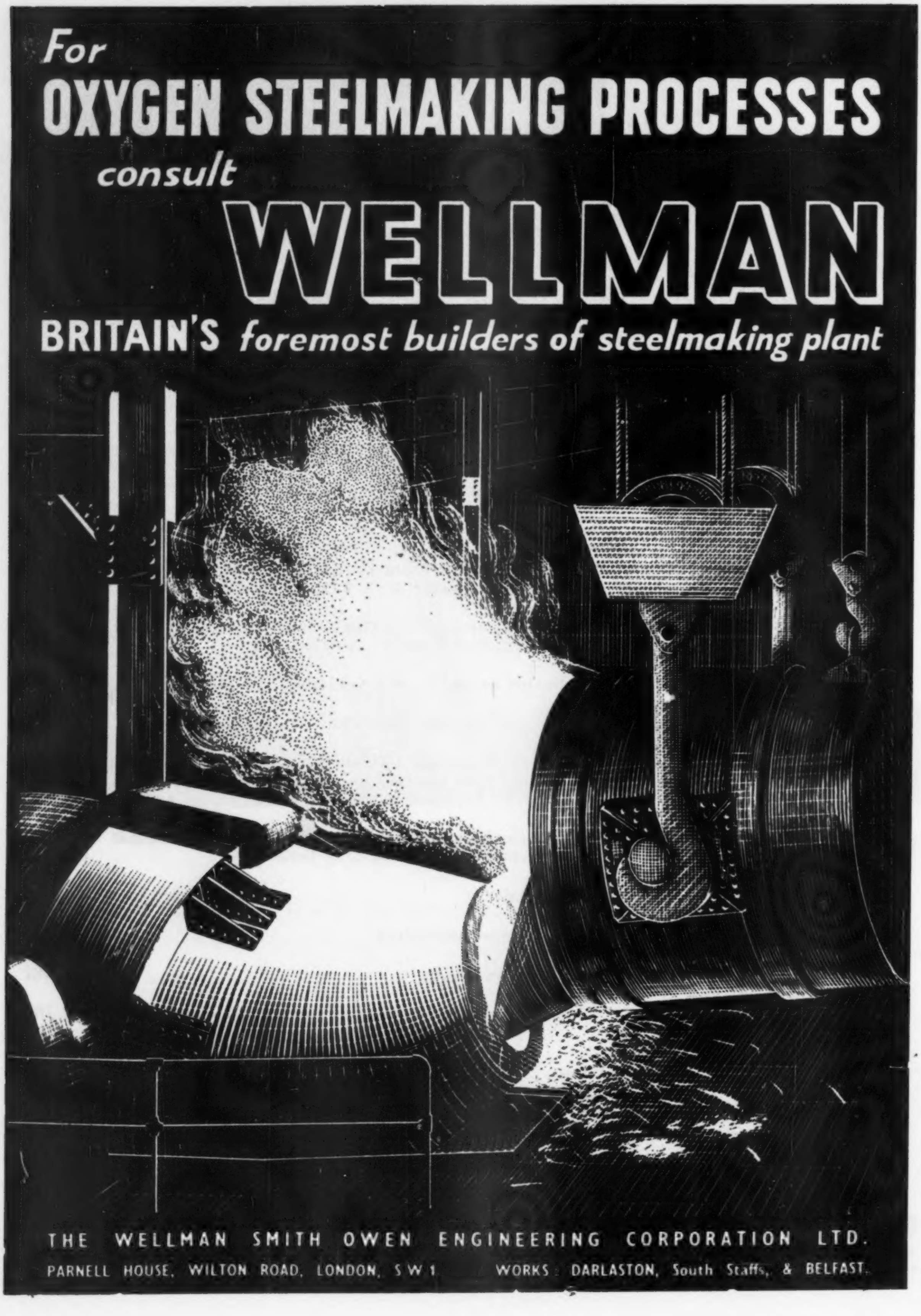
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METALLURGIA

THE BRITISH JOURNAL OF METALS
INCORPORATING THE "METALLURGICAL ENGINEER"

April, 1960 Vol. LXI. No. 366

Co-operation in Lead and Zinc

THE importance of international co-operation was stressed by Mr. R. Hendricks, executive vice-president of The Consolidated Mining and Smelting Co. of Canada, Ltd., in a talk on "Recent Developments in World Lead and Zinc Markets," presented at a joint session of the American Zinc Institute and the Lead Industries Association, held at St. Louis early in April. Lead and zinc ores are unevenly distributed throughout the world, so that some countries, such as Africa, Australia and Canada, have sufficient high-grade ore to allow production of much more than they can use, whilst others, such as the United Kingdom, India, Japan and the United States, are unable to provide for their consumption from domestic ores, and so are compelled to import. As a result, there is much traffic in lead and zinc in various forms from one country to another, and it is believed that this will increase. International trade is thus an integral part of the lead-zinc industry, and international co-operation is essential for its well-being. "This latter fact," said Mr. Hendricks, "is receiving growing recognition, and I submit that the most important single international development in the industry in recent years is the increase in co-operation that has been brought about."

For many years there has been considerable freedom between producers in exchange of technical information concerning methods of mining, concentrating, smelting and refining, and there appears to be a possibility of the Russians opening their lead and zinc mines and smelters to visitors on a reciprocal basis. "I believe," continued Mr. Hendricks, "that such exchange on the technical level is beneficial, not only for the direct results, but also in a broader sense."

As a further instance of international co-operation, there has recently been set up the International Lead-Zinc Study Group, organised under United Nations sponsorship and open to any government which is a member of the United Nations. At present it numbers twenty-five governments, representing all continents and accounting for more than 80% of estimated world production, consumption, and trade of lead and zinc, ore and metal. The Group functions as a multilateral consultant, and is authorised to make studies, arrange for statistics, consider possible solutions to special difficulties, and submit reports to member governments which may include suggestions or recommendations: the Group has no regulatory authority. One of its main interests is the problem of international market instability for these metals arising from the special circumstances in which they are produced, marketed and consumed. The Study Group is primarily a forum through which the members can seek and obtain more information and a better understanding of the outlook of others.

Even trade associations which began by being more or less national in character have now acquired an international look. The American Zinc Institute now has many associate members representing an important part of the industry outside the United States. Again, the Zinc Development Association, with headquarters in London, formerly obtained its support from the United Kingdom, Australia and Canada, and was mainly concerned with promoting the use of zinc within the United Kingdom, although its literature was also circulated in Canada and Australia. Recently Z.D.A.'s interests have spread to Europe, where there are a number of parallel co-operating associations. Even more recently, a number of U.S. firms have been supporting the Association and participating directly in its activities. Associated with the Z.D.A. are affiliated groups, such as the Hot Dip Galvanizers Association and the Zinc Alloy Die Casters' Association, aimed at furthering particular uses of zinc. In these too European co-operation is prominent; the European Pressure Die Casting Committee, for instance, is holding an international conference at Stresa, Italy, next month, and the European General Galvanizer's Association is planning an international conference on galvanizing to be held at Interlaken, Switzerland, in 1961.

Co-operation is often more readily achieved in the face of adversity than when conditions are easy, and the competition to lead and zinc provided by such vigorously promoted materials as aluminium, stainless steel and plastics had much to do with the setting up in September, 1958, of the A.Z.I.L.I.A. Expanded Research Program with the broad international backing. In presenting a statement of progress recently, Dr. S. F. Radtke, research director of the Program, drew attention to the marked success achieved in two important research areas. The development of new and improved systems for plating zinc-base die castings to provide a service life much longer than that formerly achieved, has contributed markedly to the retention of the market for die castings. The second important success concerns zinc lithographic printing plates which are subject to competition from at least four other materials. Co-operation between zinc rollers and the Lithographic Technical Foundation has developed a new graining system, and allowed the industry to evaluate a superior new zinc alloy, and to develop new surface treatments, which, it is hoped, will enable the industry to win back lost ground.

Some measure of the competition to be faced is provided by two new plastics developed by the Du Pont Company. After twelve years of research and development at a reported cost of $6 million, production is planned of Dycril, a new plastic printing plate material which will compete with zinc in lithography and photo-engraving. The other new product, Delrin, is threatening the zinc-base die casting: ten years' work and some $42 million are reported to have gone into the development of this material. Competition such as this and parallel thrusts from other materials make whole-

nearted co-operation essential if zinc and lead are to hold their own in world markets. Moreover, it is not enough to prosecute successfully a programme of research work, there must be that follow-up which will ensure that the results are exploited as quickly as possible and translated into highly competitive commercial products.

After considering in some detail the ups and downs of zinc and lead consumption in various parts of the world, and some of the economic factors influencing that consumption, Mr. Hendricks put in a plea for the consumer. " For too long industry and government alike have overlooked the importance of consumers. At the various meetings and hearings when the needs and claims of different interests are presented and action urged for their safeguarding, seldom does anyone represent consumers or put in a claim for them. I suggest that it is time that this omission was rectified, for our own well-being if for no other reason. After all, the major task facing our industry is to stabilize and increase world consumption of lead and zinc, and this will need careful attention to the interests of consumers.

" One of the prime requirements will be to see that adequate supplies of good quality metal are available at reasonable prices. Producers must keep the supply of metal coming out in line with market requirements, in poor times as well as good. We must do our best to maintain reasonable and stable prices, and these cannot be considered solely on the basis of local costs. Competition must be recognised, particularly from alternative materials. Unduly high prices may give temporary benefit, but experience has shown that in the long run they are detrimental to marketing. On the other hand, very low prices, insufficient to maintain a satisfactory level of world production and good quality, are also harmful."

" As compared to conditions a few years ago," Mr. Hendricks concluded, " there has been definite improvement in the general health of the industry, and there are many signs of encouragement. At the same time, as we are now entering the 1960's, there are still many problems facing us. International co-operation has greatly helped the lead-zinc industry in its technical problems and its broader marketing problems. With continued sincere and progressive international co-operation, I believe that our industry can look forward to a bright future, in which our products will find ever-increasing applications in the service of mankind."

Personal News

MR. W. PADLEY, C.M.G., O.B.E., has been appointed to the Board of Metal Industries, Ltd. Mr. Padley will relinquish his position as managing director of Brookhirst Igranic, M.I.'s big electrical subsidiary, and will be succeeded in that post by MR. A. B. VICKERY, O.B.E.

STEEL, PEECH AND TOZER, a branch of The United Steel Cos., Ltd., have announced a number of staff changes. MR. M. THOMAS has been appointed assistant works manager (services) and MR. E. HOUGHTON assistant works manager (special duties): Mr. Houghton will be primarily concerned with manning problems arising from the conversion from open hearth to electric arc furnaces in the Templeborough melting shop. MR. D. R. B. BURDASS is now personal assistant to the general works manager, and MR. D. REILLY has joined the company as cold rolling mill manager. Mr. Reilly was until recently with Guest, Keen and Nettlefolds, Ltd., and will be responsible in his new capacity to the works manager, Ickles, for both the existing and new cold rolling departments. MR. G. D. JORDAN, who has been senior engineer in United Steel's department of operational research and cybernetics since its formation, has been appointed Templeborough melting shop project secretary, responsible to the chief engineer. MR. J. HEWITT, of the research and development department of United Steel, is now senior research metallurgist, responsible to DR. B. B. HUNDY, chief research metallurgist, and MR. J. RAVENSCROFT, formerly with the British Iron and Steel Research Association, has joined the company as arc furnace research assistant, responsible to MR. R. S. HOWES, manager of the Rotherham melting shop.

CAUSEWAY REINFORCEMENT, LTD., have appointed MR. T. C. LEACH to be their representative in the five Midland counties for surface armouring and multi-purpose trestles. MR. C. T. WITHY is to represent the company in Lancashire and Yorkshire.

MR. J. SAMUELS, who, as a member of the board of Winston Electronics, Ltd., has been purchasing director for several years, has now been appointed works director in charge of production.

To cope with the pressure of increasing sales and technical enquiries, West Instrument, Ltd., have opened a new sales and service department in Sheffield and appointed more representatives at their London, Manchester and Birmingham offices. MR. I. WYLIE has been appointed manager of the new office covering the Sheffield area. The address is White Buildings, Fitzalan Square, Sheffield, 1 (tel.: Sheffield 22461). MR. R. W. NEWLAND has joined the Manchester office, MR. G. J. CARMICHAEL the Birmingham office, and MR. D. A. GOSWELL, the London sales and service branch.

IN a reorganisation of the executive management of C. & L. Hill, Ltd., MR. E. J. BOAG, who was previously works manager of the Phosphor Bronze Co., Ltd., has been appointed director and general manager of C. & L. Hill Ltd. and its subsidiaries. MR. W. A. BANNISTER becomes sales director and MR. L. E. MORRIS continues as works director.

ENGLISH STEEL CORPORATION, LTD., announce that DR. C. J. DADSWELL relinquishes the office of managing director of Davis & Lloyd (1955), Ltd., and is appointed chairman, and MR. C. MUIRHEAD, O.B.E., takes over the office of managing director of Davis & Lloyd (1955), Ltd. Davis & Lloyd (1955), Ltd., is a wholly owned subsidiary of English Steel Corporation carrying out the design and sale of specialised types of cast steel railway bogies. Dr. Dadswell is a director of English Steel Corporation Ltd., and Mr. Muirhead a special director.

MR. W. K. WHITEFORD, formerly of Toronto and now President of Gulf Oil Corp., Pittsburgh, has been elected a member of the advisory committee of The International Nickel Co. of Canada, Ltd. The Committee consists of persons elected by the board to assist it and the officers in the formulation of basic policies.

The Brittle Fracture Problem and the Load Carrying Capacity of Structures

By L. E. Benson, D.Sc., F.I.M. and
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The problem of brittle fracture of steel is considered in relation to strength. A component or structure may break with a brittle-looking fracture if overloaded, but this does not necessarily imply a lack of either strength or ductility and it may therefore give satisfactory service under normal loads. Information on the load which a structure can safely withstand would be very much more useful than the results of conventional notch impact tests which measure only the energy absorbed in breaking a small specimen with a standard form of notch. Published work, supported by a series of notch tensile tests, shows the general form of the strength-temperature relationship, but considerably more testing is required to assess the dependence of strength on material, temperature, size and notch geometry.

In recent years there have been a number of spectacular failures of large welded structures, such as ships, oil storage tanks and bridges.[1, 2] These failures were characterised by extensive fractures which occurred at low levels of applied stress, and which had a brittle appearance, although the materials showed normal ductility in standard tensile tests.

As the result of extensive investigations the conditions which brought about these failures are now reasonably well understood, and the significance of this type of failure being experienced with welded and un-stress-relieved structures is plain. It is also clear, however, that failure of the brittle type is possible with welded structures that have been stress relieved[3] and with components and structures that have not been welded at all.[4]

Under these conditions the strength, or load carrying capacity, of the component or structure may be greater or less than the strength indicated by the tensile strength of the material of which it is made, the effect being governed mainly by :—

(*a*) some property of the material itself ;

(*b*) the temperature at which the load is applied ; and

(*c*) the size of the part, and the size and geometry of stress concentrating features.

With regard to (*a*) no standard test has yet been devised which enables the relative strength of different materials in this special sense to be compared. The significance of (*b*) and (*c*) is that, for a given material, it is these factors which appear to determine whether a concentration of stress in a component can be accommodated by local yielding or whether it will cause fracture. Very little has been done, either to measure quantitatively the effect of size and geometry in this connection, or to produce the sort of systematic data from which general trends can be deduced.

Because of the general interest to engineers of the brittle fracture problem it seemed worth while reviewing very briefly some of the points that have been established, discussing certain aspects of the problem on which there seems sometimes to be a little confusion, and reporting the results of some laboratory experiments designed to indicate how strength may be expected to vary in relation to size and geometry as well as temperature.

Welded Non-stress-relieved Ship and Tank Failures

The explanation of the cracking of ships and tanks and other welded structures, sometime with catastrophic results, involves recognition of the following factors :—

(*a*) Marked stress concentrations, either due to design features such as sharp hatch corners or defects such as initial welding cracks, impair the ability of the material to yield and may reduce the general level of stress at which cracking starts. This is consistent with the observation that brittle fractures in welded parts always start at a stress concentrating feature of some kind. This is quite apart from any effect due to welding stresses.

(*b*) Residual welding stresses and applied stresses tend to be additive (though not strictly so if the yield point is exceeded), and failures at low applied stress can in many cases be attributed to the existence initially of a high residual tensile stress at the point of initiation of failure.

It is well known that longitudinal residual stresses in and adjacent to a weld seam can be at least as high as the yield stress for the material as normally determined. Kennedy[5] carried out wide plate tests with specimens $36 \times 36 \times 1$ in. thick, which had a butt weld down the middle and a bad artificial notch in the form of a transverse saw cut. He found that, if residual welding stresses were removed by suitable heat treatment, applied stresses of yield point magnitude were required to initiate failure, even at a temperature as low as $-56°$ C. Benson showed, in the discussion of a paper by Wells,[6] that when similar un-stress-relieved wide plates failed at low applied stresses, the real stress at the origin of failure was of about yield point magnitude when residual welding stresses were taken into account. A plot of the points relating real stress locally at the origin of failure against testing temperature is shown in Fig. 1. A feature of interest concerning these results is that whilst some of the plates contained comparatively blunt and small saw cut notches, failure in other cases was initiated by sharp cracks several square inches in area. These results will be considered again later.

(*c*) Whilst it takes a fairly high nominal tensile stress, often of yield point magnitude, to initiate a brittle fracture at a bad notch, once this brittle fracture has been started, Robertson[7] has shown in some brilliant experi-

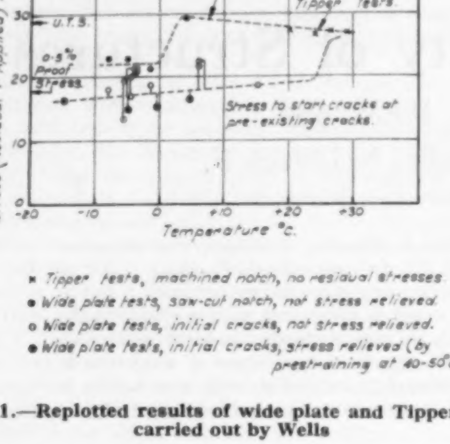


Fig. 1.—Replotted results of wide plate and Tipper tests carried out by Wells

ments that it can continue to propagate at a comparatively low stress level, e.g. 3–5 tons/sq. in. for mild steel. It is this low propagating stress that explains the very extensive nature of some cracks which have initiated close to a weld and then extended a long way from it. The stress temperature relationship for propagation, however, is critical, and Fig. 2 shows the relationship for one sample of mild steel plate as reported by Robertson.[7]

Brittle Fracture in Relation to Normal Mechanical Properties

Before considering the subject of load carrying capacity further it is perhaps as well to comment on certain factors relating to the brittle fracture problem about which erroneous assumptions are sometimes made.

Brittle Fracture v. Tensile Strength

It appears sometimes to be thought that a brittle fracture implies that there has been a lack of strength. This is not a fair assumption. It is disproved by the results of notched tensile tests of the type employed by Dr. Tipper.[2] Fig. 3, which is from a paper by Wells and his associates,[8] shows some Tipper test results as a plot of tensile strength *v.* temperature, and indicates that at the higher temperatures the fractures are ductile but at the lower temperatures they become brittle with a fairly rapid transition. The tensile strength increases steadily as the temperature falls, and continues to increase without irregularity even after the fracture has become brittle. Similar results have been obtained by Dr. Tipper on many different samples of steel ship plate. Such results show clearly that a component which breaks with a brittle fracture is not necessarily weak, and may be just as strong or even stronger than one which breaks with a ductile fracture.

It is just as well that brittle fracture does not necessarily imply weakness, because engineering structures, particularly in the realm of heavy engineering, contain many examples of parts designed to normal engineering standards, which are, nevertheless, of such size and shape that they may show a brittle fracture if overloaded to the point where failure must occur. Many examples have been observed as the result of accidents, e.g. rotor forgings, large shafts, bolts, press columns, and stern frames, in addition to welded structures.

It is true that under some conditions brittle fracture is accompanied by a loss of strength in the sense that cracking or fracture occurs at a nominal applied stress lower than the nominal breaking strength of the material as determined by an ordinary tensile test on a plain test specimen. For example, as already mentioned, failure occurred at stresses of yield point level, or about half the normal tensile strength, in tests by Kennedy[5] on stress relieved wide plates. In engineering structures such loss of strength is one of the things that is normally taken care of by an empirical factor of safety and, fortunately, disastrous weakening seems to be a risk only under rather extreme conditions, represented by a high concentration of stress and relatively " brittle " material (" brittle " here referring to a characteristic that as yet we do not know how to define, nor to measure properly, and which varies with temperature). As an example of such disastrous weakening, some alloy steel forgings in the United States[4] failed, under conditions which should have been safe, because " hair cracks " or other defects at the origin of failure provided particularly sharp notches in a highly stressed region.

The failure of welded ships and oil storage tanks can be discounted as examples of disastrous weakening due primarily to high stress concentration at notches, because they were not stress relieved, and residual welding stresses played a major part in bringing about failure. We have yet to hear of an authentic case of an effectively stress relieved welded structure that has failed seriously at normal service or test loading conditions.

Brittle Fracture v. Ductility

Another fallacy to which some people appear to subscribe is that brittle fractures are associated with negligible ductility. The degree of plastic distortion in the material adjacent to a brittle-looking fracture in steel can be a very variable quantity, and although it is sometimes quite small, it is frequently a good deal more than many people suspect. Fortunately it is generally adequate to accommodate the local stress concentration effects met with in normal engineering practice, so that there is no loss in strength.

In the Tipper tests quoted above (Fig. 3), even where the fracture is described as completely brittle in appearance, the ductility was sufficient to accommodate the stress concentration at the notches, otherwise the

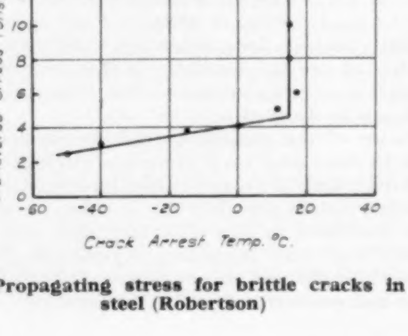


Fig. 2.—Propagating stress for brittle cracks in mild steel (Robertson)

breaking strength would have been reduced. There is no break in the curve at the ductile/brittle transition, the position of which is indicated.

An even more striking case is illustrated in Fig. 4, which shows the fractures of two tensile test pieces broken at room temperature and representing two medium carbon steel forgings made from the same cast of steel. In appearance one fracture is brittle and the other fracture tough, yet the brittle fracture specimen showed 15% elongation and 23% reduction in area. The only significant difference between the forgings is that one was a little harder than the other. If both forgings had been tempered to the same U.T.S. the fractures would have been similar. This behaviour is not exceptional with large carbon steel forgings and castings, though it may be surprising to anyone whose experience is limited to mild steel.

Brittle Fracture v. Notch Impact Value

A great deal of experimental work has been done in which the results of notched impact tests of the Charpy or Izod type, or transition temperatures based on such results, have been used as a measure of brittleness.

It is recognised that notch impact tests have value in comparing one steel with another and, in the absence of a better test, they can usefully be incorporated in specifications along with other tests as a check that the material concerned is not abnormal.

Impact tests can also play a useful part in brittle fracture investigations, but their usefulness is severely limited and they may not be as important as sometimes assumed for the following reasons :—

(*a*) Notch impact tests measure the total energy absorbed in fracturing the specimen. In some cases the greater part of the total energy is absorbed after fracture has commenced. The engineer is not interested in this ; his primary concern is to know what his materials and structures will stand *without* failing.

(*b*) Notch impact tests do not give an answer in terms that an engineer can employ in strength calculations. He requires an answer in terms of safe stress and strain rather than energy absorbed in failure.

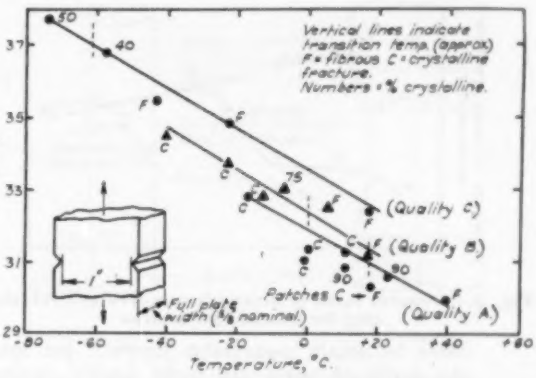
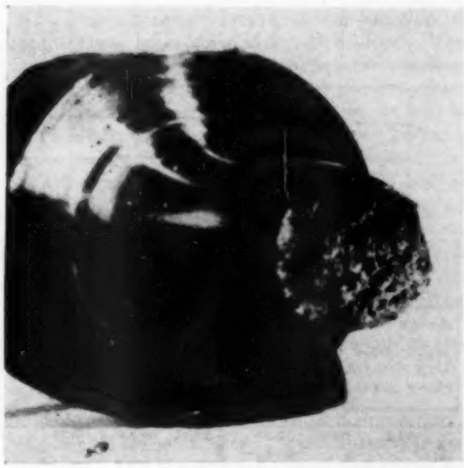


Fig. 3.—Transition temperatures of three qualities of mild steel in relation to the strength of notched tensile test pieces as shown by Tipper tests

(*c*) Whilst the effect of temperature is taken account of in determining "transition temperatures," this is only one variable. The behaviour of a notched structure is also determined to an important extent by other variables, particularly the degree of stress concentration. Size is also important and large structures must be expected to have a different transition temperature from small specimens. The effect of these variables cannot be inferred from conventional notch impact data.

(*d*) Many stressed components give satisfactory service although they are made from steels which have very low impact values at room temperature. The Izod figure for large medium carbon steel forgings is frequently in the range 5–10 ft. lb. yet it is not even considered necessary to do routine tests. Case-hardened parts are even more brittle, the normal Izod figures being almost zero (Table I reproduces[9] the results of some case-hardened impact test pieces which had blunt notches in



←Ductile Fracture

U.T.S.	45 tons/sq. in.
El.	22%
R.A....	40%

Analysis

C	0.51%
Si	0.22%
Mn	0.84%

Brittle Fracture→

U.T.S.	49 tons/sq. in.
El.	15%
R.A....	23%

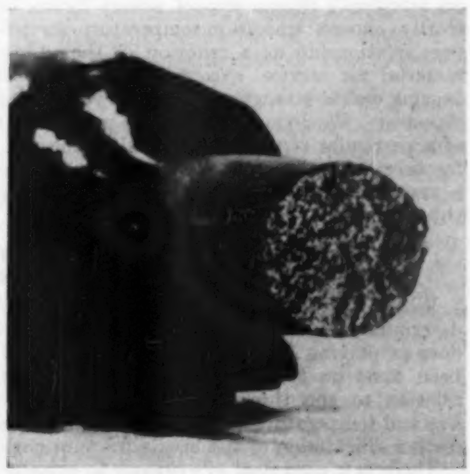


Fig. 4.—Ductile and brittle tensile fractures of medium carbon steel

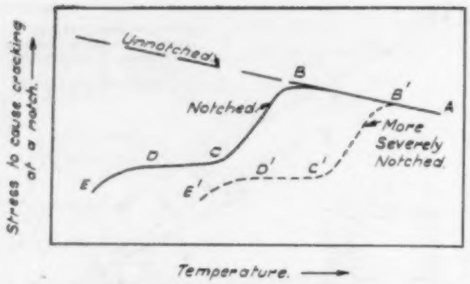


Fig. 5.—General form of strength-temperature relationship for notched structures

order to obtain measurable figures), yet many case-hardened parts are quite highly stressed. Consider the abuse which the gears in some motor-cycles and motor-cars receive. We have examined many case-hardened traction gears after service and never seen a brittle tooth fracture, except where fatigue cracks have developed first through overloading.

(e) The energy absorbed in fracturing a notched test piece is no indication of strength, and may well be related inversely to strength. It has already been shown (Fig. 3) that a change from ductile to brittle fracture brought about by a decrease in temperature can result in an increase in strength, but it would be accompanied by a decrease in energy absorbed during fracture. It has also been shown already that brittle-looking fractures are not necessarily associated with negligible ductility, and it therefore follows that low impact energy absorption does not necessarily imply negligible ductility.

In view of the above considerations, there is justification for doubting whether notch impact tests measure the characteristic property in which we are really interested when comparing steels in regard to their suitability for service in structures having stress concentrating features. At best, notch impact tests enable steels to be compared on a basis that is not directly applicable to any particular design of structure.

It would seem irrational to adopt fracture appearance, an arbitrarily chosen impact energy figure, or an arbitrarily chosen transition-temperature/service-temperature relationship as a criterion of the suitability of a material for service, except in relation to other data bearing on the strength or load carrying capacity of the structure. We do not know how to predict the behaviour of a particular structure in regard to the risk of brittle fracture unless we make *ad hoc* tests to simulate the actual full scale conditions required to be met employing the actual steel.

Load Carrying Capacity in Relation to Geometry, Size and Temperature

Whilst a great deal of effort has been expended on the determination of "transition temperatures" and other data employing notch impact tests, very little work has been done on the direct measurement of strength in relation to the three important variables—geometry, size and temperature—and such data are essential to the further elucidation of the brittle fracture problem.

There is a good deal of evidence to suggest that for a notched tensile test specimen, the general form of the

TABLE I.—IZOD TESTS ON CASE-HARDENED TEST PIECES
(notch: semicircular, $\frac{1}{16}$ in. radius; case: 0·05 in., case-hardened after machining)

Treatment		Energy Absorbed (ft./lb.)	
Quenched	Tempered	C Steel	2% Ni Steel
780° C.	—	2, 2, 3, 2	3, 2, 3, 2
780° C.	200° C.	2, 3, 3, 3	3, 3, 3, 3
880° C., 780° C.	—	3, 2, 2, 2	3, 2, 2, 2
880° C., 780° C.	200° C.	2, 2, 3, 2	5, 4, 4, 4

relationship between strength and testing temperature is of the type shown by curve *ABCDE* in Fig. 5. The precise form of the curve is determined mainly by geometry, by size and by the characteristics of the material being tested.

An important feature of the relationship is that, as temperature is reduced, a maximum strength point *B* is reached below which the ductility of the material is insufficient to accommodate stress concentrations completely, and strength falls accordingly, following the curve *BCDE*.

With mild steel a marked change in slope occurs in the region *CD* which is probably associated with the pronounced yield which characterises mild steel. As shown in the experimental results reported later (Fig. 9), the change in slope is less pronounced with harder steels and may presumably be absent with some steels. If conditions are made more severe by making the notch sharper or the test specimen larger, the strength curve is moved to the right as shown by *AB'C'D'E'*, so that the maximum strength point *B'* occurs at a higher temperature.

Some Published Data on Load Carrying Capacity

(*a*) We have already seen that Tipper notch tensile tests can show a steadily increasing strength as testing temperature falls. The Tipper curves of Fig. 3 correspond to the straight portion *AB* of the strength-temperature relationship indicated in Fig. 5.

(*b*) Robertson[10] showed that the strength of notch

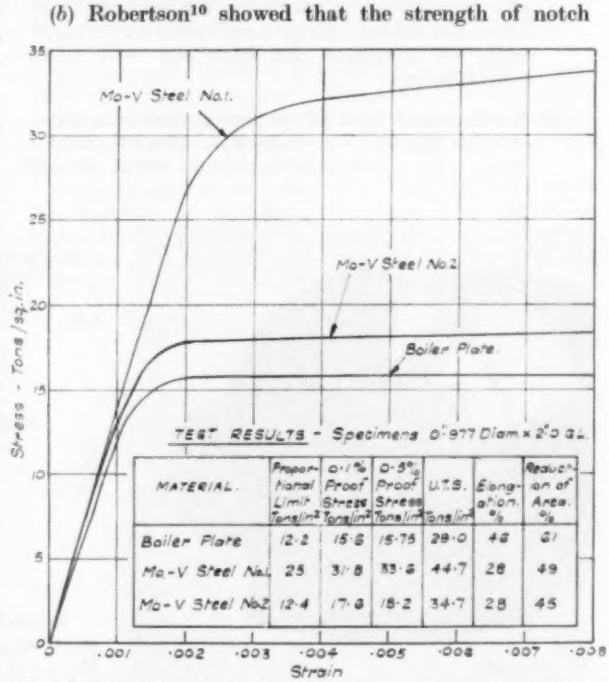


Fig. 6.—Tensile tests on plain specimens at room temperature

tensile test pieces does not rise indefinitely as temperature falls. Working with three different grades of mild steel, he used Tipper test pieces having notches ½ in. deep × 0·010 in. root radius, and also test pieces which were notched by introducing very sharp fatigue cracks. He found that, after reaching a maximum value, the strength fell off as indicated at point *B*, Fig. 5, and he found also that point *B* occurred at a higher temperature with the sharp fatigue cracks than with the 0·010 in. radius Tipper notches. With the dimensions of test pieces employed, the tensile strength was not disastrously reduced by reducing temperatures down to –45° C., and no failure was produced with a stress of less than 20 tons/sq. in., i.e. about two-thirds of the normal U.T.S. of the material. There was a marked flattening of the strength-temperature curve in the region *CD* (Fig. 5), indeed the curve sloped in the reverse direction over a limited range of temperature.

(*c*) Reference has already been made to wide plate tests on mild steel carried out by Wells,[6] and to Fig. 1, which presents the results of his tests after making due allowance for residual welding stresses. Since these residual welding stresses could only be estimated roughly, no great accuracy can be claimed for some of the points plotted, but others are quite definite because they refer to specimens which were either stress relieved by prestraining at 40 – 50° C., or which contained primary cracks extending well beyond the zone of high residual stress. The results of a few Tipper tests which Wells carried out on the same material have been added to Fig. 1 to provide a more complete picture.

The general trend of the results can hardly be other than that indicated by the two curves shown in Fig. 1, which are of the general form shown in Fig. 5. The points associated with the upper curve refer to initiation of cracks from relatively blunt notches, either on wide plates with saw cuts or machined Tipper test pieces.

The points associated with the lower curve refer to the initiation of cracks from pre-existing cracks up to 6 in. long which occurred spontaneously on welding or on application of a prior very light load. Such primary cracks provide very sharp transverse notches, which are, of course, much sharper and longer than machined notches or saw cuts. Nevertheless, it will be seen that even when these cracked plates were pulled at temperatures below 0° C. (25–30° C. below the Charpy ductile/brittle transition temperature) failure only occurred at yield point stress and when some general plastic strain had occurred in addition to the elastic strain corresponding to that stress. As in Robertson's work, the maximum strength point (point *B* in Fig. 5) occurs at a higher temperature on the lower (sharp notch) curve than on the upper (blunt notch) curve.

Data reported by Kennedy[5] relating to stress relieved mild steel wide plate tests is consistent with the presentation of Wells' data shown in Fig. 1.

(*d*) The shape of the more or less vertical portions of the curves shown in Fig. 1 was assumed from incomplete evidence, but the steep slope of the curve in this region has since been confirmed by Japanese workers.[11]

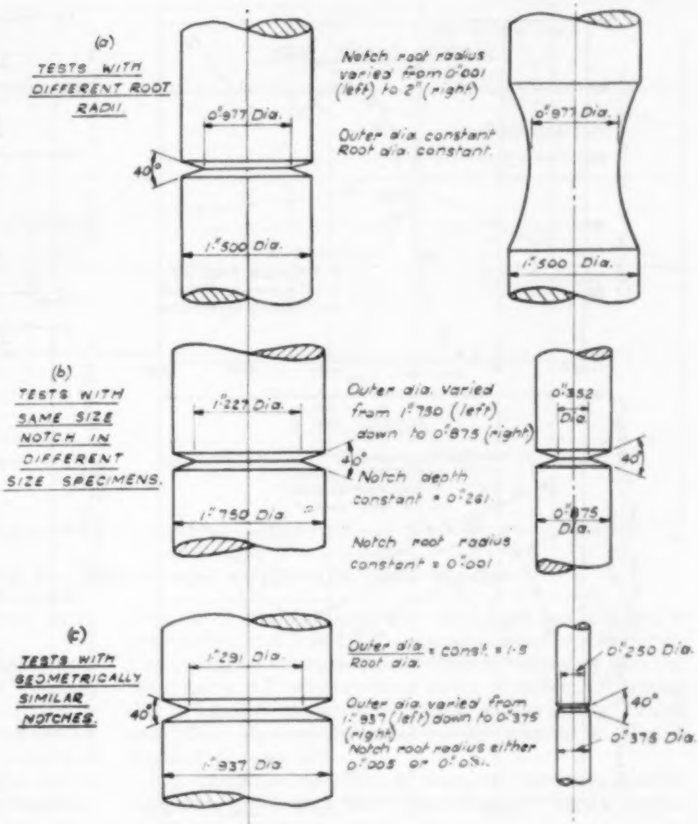


Fig. 7.—Specimens for notch tensile tests

(*e*) It is interesting that a curve of similar form to that shown in Fig. 5, with a point of maximum strength, a steep fall in strength at lower temperatures, and an abrupt change of slope in the region of the yield point, has recently been reported[12] for unnotched tensile test pieces of mild steel at temperatures in the region of –100° C. to –200° C. Although there was no machined notch it appears that at such low temperatures stress concentration effects are introduced by minor defects in the steel, by the different orientation of the lattices of adjacent crystals, and by the formation of micro-cracks.

Experimental Data on Load Carrying Capacity

To enable an assessment to be made of the strength of a structure under service conditions, a background knowledge is desirable of the general trend of the relationship between strength and severity of conditions, "severity" in this sense being not only a function of temperature, as in Fig. 3, but also of geometry and size. To supplement published information such as that described above, and to try to indicate the general trend of the strength-temperature relationship, a series of notch tensile tests has been carried out in which the variables introduced included :—

(*a*) material,

(*b*) temperature,

(*c*) depth and sharpness of notch, and

(*d*) size of test piece.

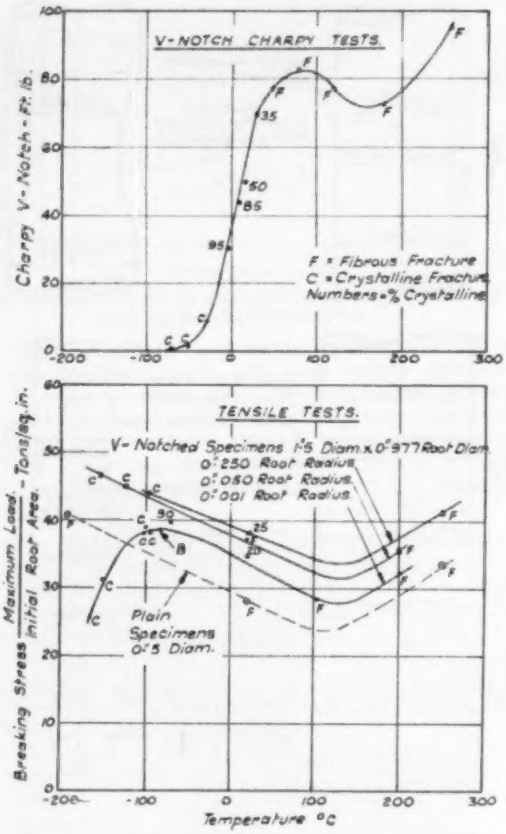


Fig. 8.—Charpy and tensile tests on boiler plate

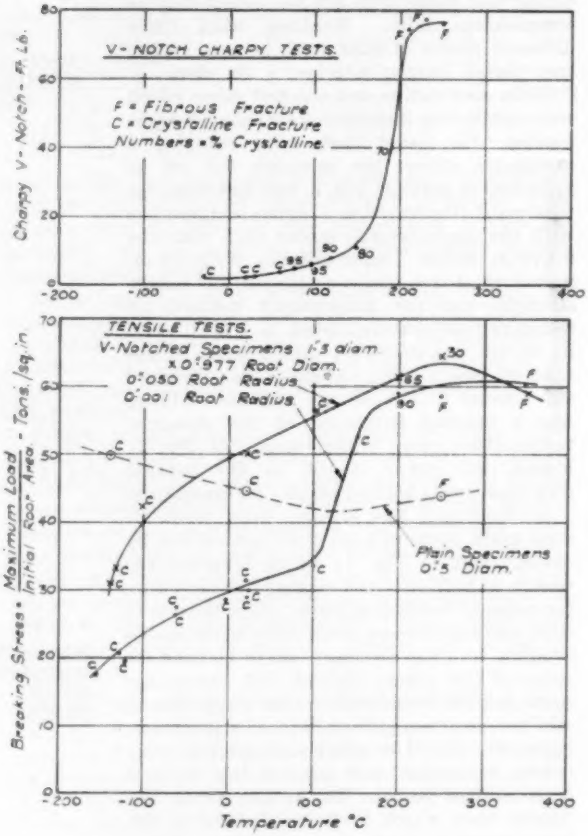


Fig. 9.—Charpy and tensile tests on Mo-V steel No. 1

Particulars of the three materials used are set out in Table II and the results of normal room temperature tensile tests are given in Fig. 6. Fig. 7 shows the form of some of the test pieces. The maximum size was limited by the capacity of the testing machine available, which could provide a maximum load of 120,000 lb. It is intended, however, to test larger specimens in larger testing machines, using maximum loads up to, say, 1,000 tons.

Figs. 8, 9, 10 and 12 show some of the results obtained. Fig. 8 shows the strength-temperature relationship for specimens cut from 3 in. thick "fully-killed" boiler plate. With very sharp machined notches, the point *B* (Fig. 5) of maximum strength was only reached with a temperature as low as −80° C., and although at lower temperatures the strength fell off as expected, at −160° C. point *C* had not been reached and the breaking strength was about 60% of that determined on a plain specimen at the same temperature. With blunter notches, even point *B* was not reached with temperatures down to −160° C. Strength was therefore higher than the normal room temperature tensile strength, except when the notch was very sharp and the temperature very low. At temperatures above 100° C. strength rises because of the effects of strain ageing. A Charpy vee notch transition curve for this material has been added for purposes of comparison.

Fig. 9 shows similar strength-temperature relationships for specimens cut from a large Mo-V steel forging. This material was chosen because, as shown by the Charpy vee notch transition curve, it is more brittle than the 3 in. thick boiler plate to which Fig. 8 refers, and it was hoped that with it the strength-temperature curve could be determined down to quite low stresses. Referring to Fig. 9, the point *B* of maximum strength for the notched specimens evidently occurs at a temperature of 200 °C., or higher, where the shape of the curve is confused by strain-ageing effects. Strength falls progressively as temperature is reduced. With sharp notches, it is evident that the strength of this material may be quite

TABLE II.—MATERIALS FOR NOTCH TENSILE TESTS

Material	Chemical Analysis (%)									Heat Treatment
	C	Si	Mn	S	P	Ni	Cr	Mo	V	
Boiler Plate	0·135	0·17	1·20	0·025	0·018	—	—	—	—	Normalised 900° C.
Mo-V Steel No. 1 ..	0·23	0·17	0·54	0·027	0·021	0·18	0·38	0·63	0·26	Normalised 975° C. tempered 675° C.,
Mo-V Steel No. 2 ..	0·22	0·24	0·55	0·037	0·026	0·28	0·13	0·53	0·25	Normalised 975° C.

low. For example, with the sharpest notch the strength at −160° C. was only 30% of the breaking strength of a plain specimen.

Fig. 10 shows for two different materials the effect of changing the sharpness of the notch, without otherwise changing the shape or size of the test piece. The sharpest notch was machined with a root radius of about 0·001 in. Whilst this represents a much more severe stress concentration than would be tolerated in engineering design, it is blunt compared with the sharp notches that may occur by accident, e.g. haircracks in forgings or contraction cracks in welds. It must be borne in mind that the shape of curves of the type shown in Fig. 10 may be different for different geometries and different temperatures.

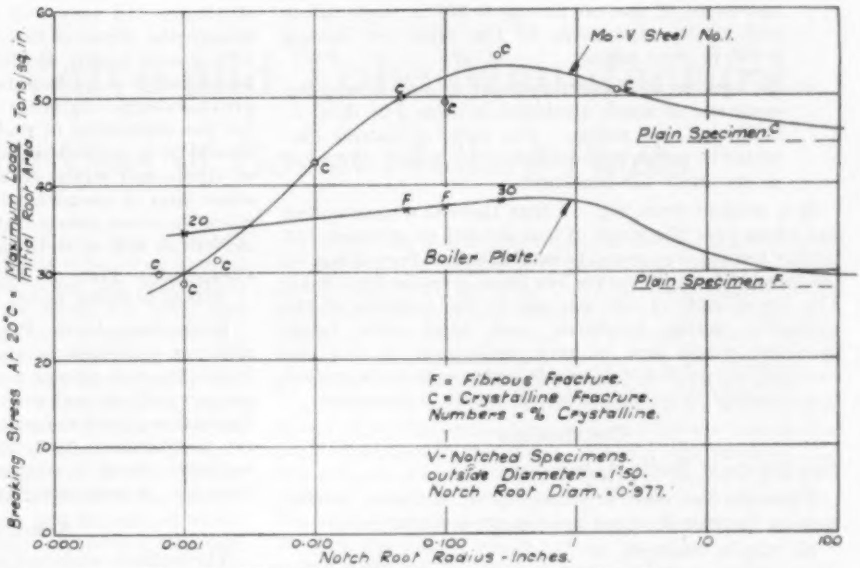


Fig. 10.—Effect of notch sharpness on tensile strength

It is instructive to note that whereas with plain specimens the Mo–V steel has a higher tensile strength and yield point than the boiler plate, it is less able to accommodate a high degree of stress concentration by yielding, so that under sufficiently severe conditions it is actually weaker. This is shown by the way the curves cross. In practice, of course, the relative strengths under brittle fracture conditions would depend on geometry, size and temperature. Another point of interest is the way in which for both materials shown in Fig. 10 the strength first increases and then decreases as the conditions are made more severe by decreasing the notch root radius. The general form of the curves is similar to the portion ABC of the curve shown in Fig. 5, which suggests that "severity of testing conditions" might be substituted for "temperature" in Fig. 5, an increase in severity meaning either a decrease in temperature or a decrease of notch root radius.

Referring again to Fig. 5, in which temperature is the variable, it can now been seen that some modification is required since AB and AB' are not necessarily coincident. Depending on whether the notches are relatively blunt or very sharp, AB' will be either above or below AB, giving rise to families of curves like those shown in Fig. 11 (A) and (B). The experimental results plotted in Fig. 8 conform to type (B).

Fig. 12 shows the effect of changing specimen size for specimens cut from a Mo–V steel forging. Two different curves are given :—

(*a*) For geometrically similar specimens all having a ratio of outside diameter to notch root diameter of 1·5. In these tests the notch root radius should have been increased progressively from 0·001R in. to 0·005R in., as the notch root diameter was increased from ¼ in. to 1¼ in. To simplify manufacture of specimens, however, two sets of tests covering the size range were carried out, all the

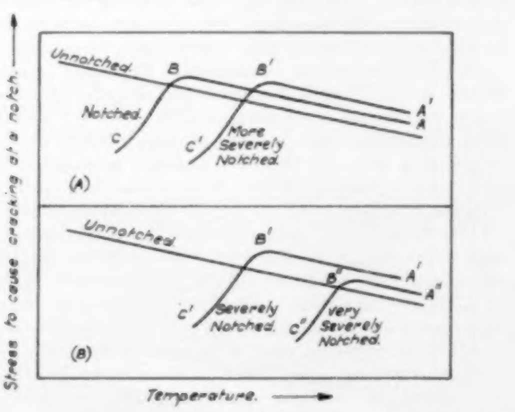


Fig. 11.—Modified forms of strength-temperature relationship for notched structures

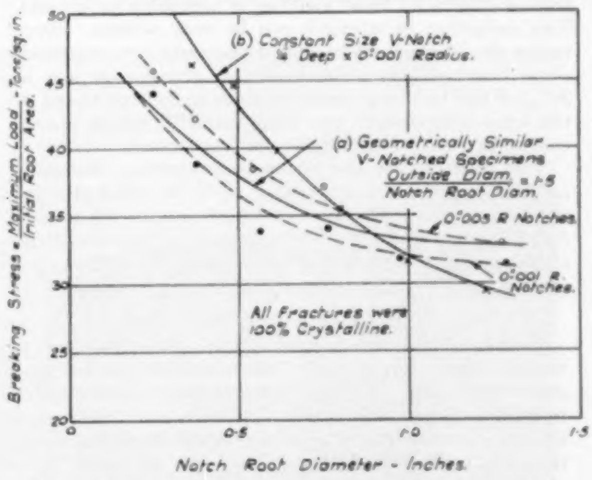


Fig. 12.—Effect of size on tensile strength at 20° C.: Mo-V steel No. 2

specimens of one set having 0·001 in. root radius and all the specimens of the other set having 0·005 in. root radius.

(*b*) For specimens of increasing size, but all having the same size of notch machined in them $\frac{1}{4}$ in. deep × 0·001 in. root radius. The ratio of outside diameter to notch root diameter fell from 2·33 to 1·4 as specimen size increased.

It is evident from Fig. 12 that there is a pronounced size effect over the range of test conditions selected, but further testing is required to establish the effect of size on the form of strength curves like those given in Figs. 8–10. The upper limit of size was set by the capacity of the available testing machines, and tests with larger machines would here be very useful and, in fact, are essential to enable notch tensile test results to be related to behaviour in service under all practical conditions.

Conclusions

Significance of Brittle Appearance

Evidence has been produced to show that a brittle-looking fracture does not necessarily indicate either

(*a*) lack of strength, or

(*b*) inadequate ductility.

The appearance of brittleness in a fracture does not necessarily indicate therefore that anything has been wrong. Indeed, many structures operating satisfactorily in service would show a brittle fracture if overloaded to the point of failure.

Factors Governing the Strength of Structures having Stress Concentrating Features

Although brittleness does not necessarily imply weakness, evidence has also been produced to show that under certain conditions a brittle fracture can occur in a general stress field substantially lower than the breaking stress of the material as normally determined.

Figs. 5 and 11 indicate that increasing the severity of the test conditions by reducing the temperature may at first result in some increase in strength, but sooner or later a maxiumum strength point is reached beyond which strength falls off sharply, presumably because the ability or the material to accommodate stress concentration effects by local yielding is becoming exhausted. This reduction in strength can be very serious. Even under the limited conditions of the tests here reported, some notched specimens broke at stresses as low as 30% of the breaking stress of plain specimens tested at the same temperature (see Figs. 8 and 9), which is even below the elastic limit of the plain specimens.

The geometry of the stress concentrating feature is an important factor governing both the strength and the temperature at which strength begins to fall off (see Figs. 8, 9 and 10). The existence of important size effects has also been demonstrated (see Fig. 12).

The Need for Quantitative Data on the Strength of Structures having Stress Concentrating Features

Following the spectacular failure of a number of welded ships, storage tanks and bridges, a great deal of effort has gone into the investigation of the brittle fracture problem, as a result of which a good deal is now known about the importance of residual welding stresses, the effect of temperature, and the advisability of avoiding notches. Evidence has been given above regarding the way in which the strength of notched structures will vary with geometry and size. Important though the whole of this information is, it deals mainly with general trends, and it is suggested that a great deal of quantitative experimental work is now required to provide design engineers with the sort of data necessary for the estimation of real factors of safety. Such data should be in terms that an engineer can use, i.e. in terms of stress and strain rather than energy of fracture or some kind of transition temperature. The data should relate to stress concentration associated with accidental defects as well as to those inherent in design features.

Comparative Tests of Different Materials in Regard to Ability to Resist Brittle Fracture

It has been shown (Fig. 10) that the relative merits of different materials in regard to their ability to resist brittle fracture are not a simple function of some inherent property of the materials but depends on the conditions they are required to meet. It is considered therefore that no standardised test has significance for the design engineer except in relation to other data bearing on the strength, or load carrying capacity of the structure.

Acknowledgments

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Machine Tool Exhibition

PREPARATIONS for the International Machine Tool Exhibition organised by The Machine Tool Trades Association and to be held between 25th June and 8th July this year are sufficiently far advanced to enable an indication to be given of its size and range. Unlike any other exhibition devoted to the display of machine tools anywhere else in the world, it is international in the fullest sense. Provided that an importing member of the Association holds an agency for a foreign machine, it may be exhibited irrespective of the country of origin.

The Exhibition will be staged at Olympia, in London, where, in the Grand, National and Empire Halls, stand space will cover more than 280,000 sq. ft. Subject to final adjustment the number of standholders at present recorded is 266. Of the total number of exhibitors, 107 will have on display British-built metal cutting or metal forming machine tools. Foreign machines will be shown on the stands of 45 importing members of the Association, and will amount to some 40% of the total exhibits, as compared with a figure of 35·5% for the last exhibition in 1956. The allied trades, producing heat treatment furnaces, welding equipment, twist drills, milling cutters, small tools, grinding wheels, jigs and gauges, etc., will be well represented. There will be 95 exhibitors of this type of equipment, and the remaining 19 exhibitors, non-members of the M.T.T.A., will be under the heading of services, e.g. the technical journals, banks, etc.

Electronic Valve and Tube Manufacture

Interesting Uses of Gas at Mullard Plants

SINCE the end of the war there has been a steady increase in the amount of gas sold to industrial users. The 770 million therms for the year ended March, 1959, is almost double the figure for 1945, and represents over 30% of the total sales of gas. About a thousand industries use gas for the carrying out of some four thousand different processes, and opportunity was recently afforded representatives of the technical press to see various gas-consuming processes in the production of electronic equipment. The occasion was a visit, arranged by the Gas Council and the North-Western Gas Board in conjunction with Mullard, Ltd., to the plants at Simonstone (nr. Burnley) and Blackburn where the Mullard company manufacture television tubes, and receiving valves and fine wire, respectively.

Television Tube Production

The Simonstone plant produces over 1½ million television tubes a year—a large part of the Group's total output. The plant, which has been described as the most modern of its kind in Europe, was completed in 1955. Since then it has expanded considerably and now employs some 1,700 people, the majority of whom are men.

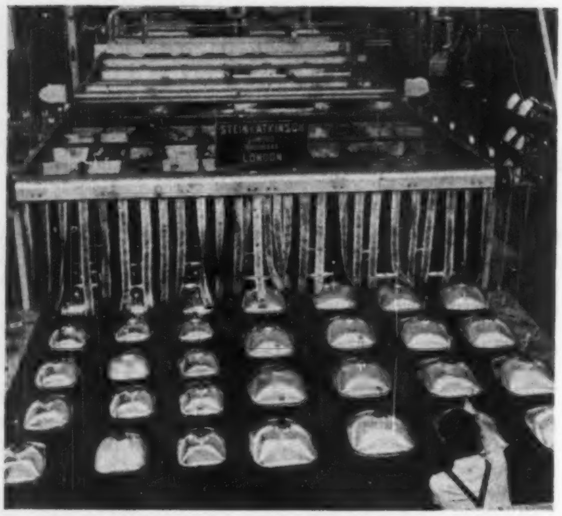
The production of television tubes begins with the component parts of the glass bulb—the cylindrical neck, the flared cone, and the rectangular faceplate. The first operation is welding the neck to the cone by means of an automatic rotary machine, of which there are six in the plant, each having twelve stations. The two parts are first pre-heated by radiant cup gas burners and gas-air burners to bring them to the correct temperature for welding, and the weld is then made by oxy-coal gas jets. Following this, the bulbs are transferred to a side sealing machine where the metal e.h.t. connection is fused into the side of the cone. The region of the seal is pre-heated by radiant cup burners and gas-air jets, and the seal is made by oxy-coal gas jets. To remove any strains in the glass, the neck and cone assembly is annealed in an oven, which also serves to pre-heat the assembly ready for the faceplate to be joined on. Meanwhile, the faceplate is pre-heated to the same temperature in another oven.

The twenty-four automatic machines for welding the faceplate to the neck and cone assembly are among the most ingenious in the plant. Gas-air jets pre-heat the two component parts, and cross-fires of oxy-coal gas jets further raise the temperature of the welding region. The actual weld is accomplished by an electric arc struck through gas flames which brings the edges of the two components up to melting point, when a perfect seal takes place. With the thick glass necessary for television tubes it is essential to heat the two components evenly to avoid strains or cracks, and to ensure this the gas jets automatically follow the contour of the faceplate, keeping a constant distance from the glass as it rotates.

The complete bulb is annealed by raising it to a certain temperature and allowing it to cool slowly, after which it is introduced to the two-mile long overhead conveyor system that runs through the entire plant, and around which all the subsequent processes are planned.

The cooled bulbs are thoroughly washed with different solutions, finishing with purified water, to make ready for the vital process of laying the fluorescent screen by means of specially designed rotary mills fitted with dispensers which automatically deliver the correct amount of buffer solution, water glass and fluorescent powder. As the bulbs travel round the mill the fluorescent powder gradually settles down through the buffer solution evenly onto the surface of the glass, where it is held by a gel-like formation produced from the different solutions. As

The Mullard television tube plant at Simonstone, near Burnley.



One of the 100 ft. Lehr furnaces for baking the fluorescent screen material on to the glass faceplate.

they complete their journey the bulbs are automatically tilted to remove the liquid, leaving the screen deposit undisturbed. The bulbs are dried by infra-red lamps and filtered air and are then given internal coatings of lacquer and graphite, the latter to form an electrical connection between the final anode and the e.h.t. connector. The bulb is then ready for laying a film of aluminium by vacuum deposition on the inside of the screen and cone. The film acts as a mirror behind the fluorescent screen, increasing the brightness of the picture ; it also prevents the screen being damaged by heavy ions during the tube's working life.

The bulbs are next passed slowly through one of two continuously operated 100 ft.-long Lehr furnaces to drive off all moisture and to bake out the unwanted constituents of the various coatings, which would otherwise make it impossible to maintain the necessary vacuum in the finished tube. This baking process also fixes the fluorescent screen firmly to the glass face. The Lehr furnaces are indirectly heated by gas fired tubes, and are the biggest single gas consuming units in the entire plant. They each have a starting consumption of 6,000 cu. ft. of gas an hour and an average hourly running load of 4,000 cu. ft. The bulbs travel through the furnaces on a 12 ft. wide conveyor belt and the whole process takes 125 minutes. The temperature through the furnaces is zoned automatically at seven points, rising from 20° C. through 420° C. and back to 25° C.

After a thorough inspection, the bulb is ready to receive the electron gun. These guns are assembled in a separate part of the factory by highly trained female operators. The component parts (over 60 to each gun) are manufactured to extremely high standards of accuracy, and a system of statistical quality control is applied throughout so that there is no possibility of faulty parts reaching the assembly operation. Oxy-coal gas jets are used in the assembly of the guns, to weld the glass support rods in the electrode assembly.

The assembled guns are checked by microscope to ensure that the spacing of the electrodes is correct. They are then washed to remove contamination before passing to the rotary sealing machines, which seal the gun, mounted on its glass base, into the neck of the bulb. Each sealing machine has twelve stations and employs radiant cup gas burners for pre-heating the region of the join and oxy-coal gas jets for the actual seal.

After this operation the tubes are evacuated whilst passing through a long heated tunnel. The heating drives off any gases present in the metal electrodes, the glass bulb, or the screen materials. At the same time, an electric current is passed through the heater to give the cathode the necessary emissive properties. These operations completed, the getter is " fired " by induced high frequency currents.

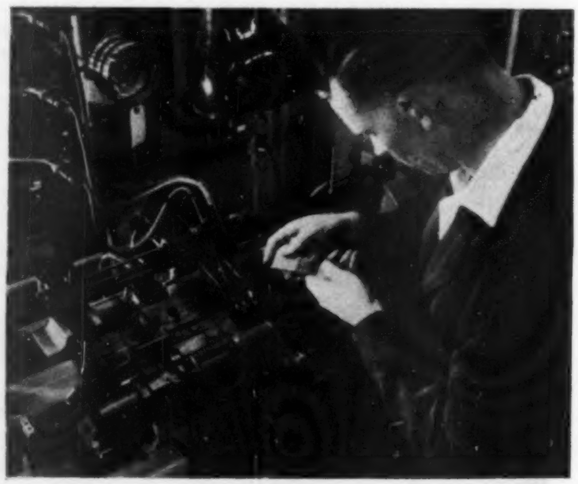
The tube is now connected to power supplies and its internal metal components are bombarded by electrons from the cathode to stabilise its performance. It is then subjected to a series of rigorous tests under working conditions to ensure its high performance and reliability.

Valve Manufacture

Established shortly before the 1939 war, the Blackburn plant employs nearly 5,000 people and is responsible for most of the Group's production of radio and television valves, turning out about a million a week. Apart from the mica spacers and the nickel used for some electrodes, all the components for the many different types produced are made within the plant from the raw materials.

The basic components of a valve are : the electrodes, comprising heater, cathode, anode and, excepting diodes, one or more grids ; the glass envelope and base ; the internal supporting members, mica spacers and insulators. These various component parts are fabricated in separate units of the plant and brought together in the large assembly bays where skilled operators build-up the complete valves.

The cathode of the valve, the electrode that provides the emission of electrons, consists of a central core of nickel tube, round which the emissive coating is sprayed. The nickel tubing is re-drawn in the plant's wire drawing unit to the very small dimensions required in cathodes, which may scale only 0·75 mm. × 0·65 mm. with a wall



Machine for welding the component lengths of the tri-metal connecting wires, comprising stiff external connecting pins, a special type of wire to form a perfect seal with the glass base, and the connecting wire between this and the various electrodes.

The reducing furnaces for converting tungsten and molybdenum oxides into metal powder.

thickness of 0·05 mm. The emissive coating, a mixture of barium and strontium carbonates in a volatile solvent, is sprayed on under careful control to ensure a coating of uniform density and thickness. Inside the central tube of the cathode is inserted the heater, or filament, consisting of a spiral of very fine tungsten wire formed into a " V " or " M " shape and coated with aluminium oxide to insulate it electrically from the cathode. The manufacture of heaters and cathodes calls for great delicacy and refinement to ensure high standards of performance and long life in the finished valves.

The grids of the valves are essentially flattened spirals of very fine molybdenum wire wound on stiff " backbones " or supporting rods. They are wound automatically on precision winding machines in lengths of about 4 ft. at a time, and each length is then cut into individual grids of appropriate size. Anodes are pressed to the required shape and size by automatic machines from nickel, nickel-plated steel or aluminised steel, depending on the type of valve.

In the assembly bays operators, assisted by ingenious mechanical aids, assemble the electrodes into a " cage," ready to be mounted on the base. The base is a small disc of glass holding the connecting pins by which the valve plugs-in to the valveholder and a corresponding number of wires to which the various electrodes are connected. Joining the stiff metal pins and the thinner connecting wires is a third length of wire which has the same overall coefficient of expansion as the glass, and which therefore ensures a perfect airtight seal where the wires pass through the glass.

The machines that make the bases automatically feed the correct number of tri-metal connecting wires into a jig. A collar of glass is placed round the wires and the whole assembly is revolved. As it rotates, gas flames soften the glass collar and the machine gently presses it into shape around the centre sections of the tri-metal wires. Special machines then cut the connecting wires into the right lengths and bond them to the exact positions for the electrodes to be welded to them.

Once the electrode cage has been joined to the base, the next steps are to seal the assembly into its glass envelope and then to evacuate the air to form the vacuum necessary to the operation of the valve. Substantial quantities of gas are used in these operations. In the first, gas jets are employed to soften the glass at the junction of the base and the envelope before the sealing machine automatically applies the correct pressure to form the joint ; and in the second for pinching off the pumping stem once the air has been removed. Twenty-four rotary pumping machines, each capable of handling thirty valves at a time are in operation.

While the valve is being pumped the metal components inside are heated by induced high frequency currents to drive out any gases which may have been absorbed during manufacture. A current is passed through the heater to reduce the cathode coating to oxide, thus forming the electron emitting surface, and induced heat is again used to evaporate the getter to absorb the last traces of gas. When the valve has been removed from the machine it is operated for a period under controlled conditions. During this period changes occur in the cathode coating resulting in improved emission.

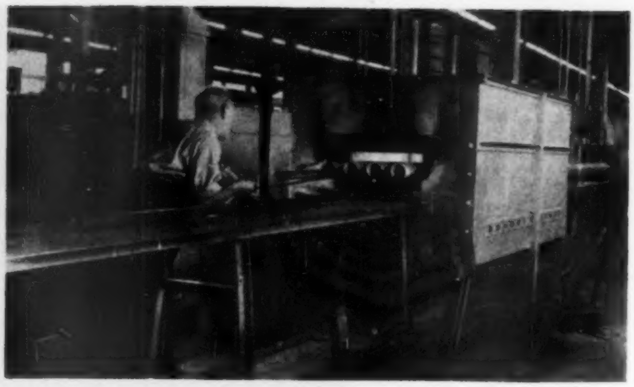
Finally, the valve is put through a succession of exacting tests covering its operating characteristics, emission, vacuum, insulation and so on. In addition to these routine tests, the quality control laboratory continually tests batches of valves selected at random from the production line. Any falling off in quality is detected at the earliest possible moment, so that steps can be taken to correct it before the trouble can assume serious proportions.

Making Fine Wire

A large section in the Blackburn plant is given over to the manufacture of tungsten and molybdenum wire. Much of this appears as grids and filaments in the valves produced by the plant, but a substantial amount is supplied to other manufacturing industries for a variety of purposes. The annual output of wire exceeds a quarter of a million miles. Most of it is extremely fine : the finest has a diameter of five thousandths of a millimetre. Like most other components of Mullard valves, the wire is made in the plant from start to finish.

Tungsten occurs naturally in the tungstates of iron, magnesium and calcium. It is the last named, known as scheelite, that is used at Blackburn. The quality of scheelite available from various world sources varies greatly, and skill and experience are necessary in its selection. Before production begins, the scheelite ore is

The sintered bars of tungsten or molybdenum are hot swaged into rods ready for wire drawing.



Furnace for intermediate annealing during wire drawing.

put through chemical tests to ensure its quality, after which it is ground continuously for several days in a ball mill. The resulting very fine tungsten oxide powder is purified chemically and placed in reducing furnaces where, in a hydrogen atmosphere, it is converted to tungsten metal powder. The reducing furnaces, which run at temperatures up to 900° C, are gas burning. Controlled amounts of special additives are introduced into the pure powder to control its crystal structure and to prevent the finished wire sagging, and the powder is then compressed into bars of convenient size. Next come the sintering processes which crystallise the compressed powder into a hard metal, ready to be made into wire. Sintering is carried out by first heating the bars in an atmosphere of hydrogen and then passing through them an electric current of 2,000—3,000 A.

The first step in converting the sintered bars into wire of the required thickness is known as swaging. Gas furnaces are used to heat the bars to white heat before they are hammered in successive stages to the form of thin rods.

The tungsten rods are drawn through a series of progressively smaller dies until they have been reduced to the thickness required. Drawing occurs in three main stages : first on a chain bench, next on large diameter drums; and finally on smaller drawing heads. In each stage a number of dies are used, carefully spaced to achieve a gradual reduction in diameter and, at the same time, to increase the strength and ductility of the wire. Hard metal dies are used in the initial stages, but as the wire becomes thinner diamond dies are necessary. Gas is used extensively throughout the drawing operations, for heating the wire before it is pulled through the dies, and also in the annealing furnaces through which the wire passes at pre-determined intervals to remove strains.

Once the required diameter has been obtained the wire is put through a series of strict tests and inspections to check its dimensional accuracy and mechanical properties. Often it is so fine that its diameter can be measured only by accurately weighing it against a standard length.

Molybdenum is obtained from molybdenite ore, the chief deposits of which are in the American continent, in Norway, and Yugoslavia. The production of molybdenum follows a pattern broadly similar to that for tungsten, with variations dictated by the chemical differences in the two ores.

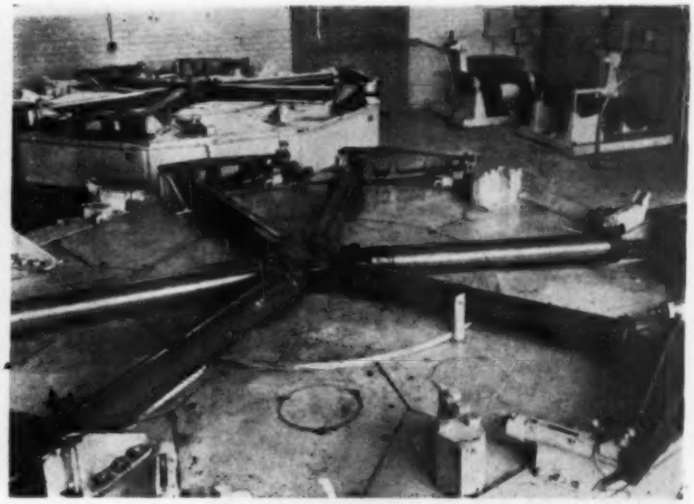
Glass Tubing Production

Glass figures so largely in the manufacture of radio and television valves that the glass-making unit at the Blackburn plant is virtually a factory in its own right, producing from the raw materials over 14,000 miles of lead glass tubing every year. The basic ingredients—red lead, sand, sodium and potassium carbonate, and additives selected to improve quality—are charged into the glass melting tanks maintained at about 1,500° C. by an oil fired furnace. From the tanks the glass flows into a gas heated holding and feeding furnace containing rotating tube forming machines. As production of glass tubing is continuous, a reliable supply of gas at the right pressure is essential to the operation of the entire glass-making unit.

Intermediate wire drawing operation using large diameter drums.

Coil Spring Research

New Laboratories Opened at Sheffield



General view of the fatigue testing laboratory showing 12½ h.p. radial machines for multiple fatigue testing of heavy hot-formed springs.

NEW laboratories for the Coil Spring Federation Research Organisation at Sheffield, which were opened on Thursday, March 24th, by Sir Harry Melville, K.C.B., F.R.S. Secretary of the Department of Scientific and Industrial Research, are among the most comprehensive of their kind in the world. They will enable research to be carried out on springs made from the finest wires to bars of up to 2 in. diameter. In addition to work on springs and torsion bars, research will be possible on materials in wire and strip form, and metallurgical investigations will be undertaken on a very wide range of ferrous and non-ferrous alloys. Included in the research programme are investigations on the influence of type of material, heat treatment and surface finish on the fatigue life of heavy duty springs; on the problem of hydrogen embrittlement in plated high tensile springs; and on the effect of non-metallic coatings on fatigue life. The importance of this work will be appreciated when it is realised that in any mechanism incorporating a spring, the spring is the most highly stressed component, and therefore the part most likely to fail if not properly designed and manufactured. Moreover, although it may only be a tiny part of a machine, its failure will bring the machine to a halt.

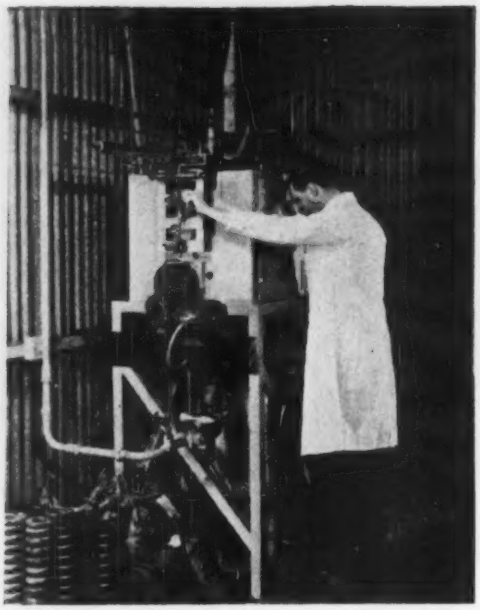
The spring as we know it—whether leaf-spring, coil spring or torsion bar—was first made in Britain about two hundred years ago. Spring-making soon became an important craft and manufacturing centres grew up in the Black Country, in Yorkshire, in Rochdale and in Redditch. These are still the most important centres, producing, for the engineering industry in general, millions of springs of all shapes and sizes, which contribute in no small measure to the reputation of British industry for quality and reliability. Only thirty years ago springs were almost entirely the product of craftsmen, being developed largely on empirical lines, but the second world war brought with it a demand for springs able to stand up to exceptionally severe conditions without risk of failure, and thus emphasised the need for a more scientific approach. This challenge was met by the setting up of the Coil Spring Federation Research Organisation, so that the industry now enjoys the benefit of the combined efforts of the craftsman and the scientist.

Although the C.S.F.R.O. came into operation in October 1945, until 1957 its investigations were carried out at the universities on an extra-mural basis. Three years ago, however, it was decided that the organisation should have its own staff and facilities, and a start was made by setting up headquarters in part of the British Iron and Steel Research Association's premises at Hoyle Street, Sheffield. Although the C.S.F.R.O. now has its own premises in Doncaster Street, Sheffield, on a site adjacent to the Hoyle Street laboratories, co-operation between the two bodies continues. By payment of a retainer fee, the Organisation is able to make use of the B.I.S.R.A. library facilities, and in a similar way B.I.S.R.A. machine shops are available for test piece preparation, etc. Nor is the link with the universities severed, as at least two of the projects in the current research programme are being carried out by university departments.

The New Laboratories

The new two-storey laboratory block is probably the most comprehensive of its kind for research into all forms of springs and spring materials. The ground floor contains laboratories for heavy-fatigue testing, general mechanical testing, experimental heat treatment and electroplating, whilst the first floor accommodation includes light laboratories (containing small static testing machines and general scientific instruments and equipment), administrative offices, and a conference room. The laboratories were built at a cost of £22,000 and new equipment totalled another £25,000.

The heavy fatigue testing laboratory is dominated by the two 12½ h.p. horizontal machines, in which the specimens are arranged radially and the load applied by means of a rotating eccentric at the centre. Each machine can accommodate nine heavy coil springs, applying a dynamic load of up to 9 tons. The rate of loading is not high, being of the order of 120 cycles per minute. Other machines in this section for fatigue testing springs of the



Shot-peening plant used for experiments on all types of springs and spring materials.

Multiple high speed fatigue testing of springs of the internal combustion engine type.

internal combustion engine type are capable of infinitely variable speeds of compression of up to 4,000 per minute. As the fatigue failure of a coil spring takes place under a torsional stress system, machines for torsional fatigue testing of spring materials are also available. A special feature of this laboratory is the sound-proofing and anti-vibration features incorporated in both the suspended ceiling and the floor.

The mechanical testing laboratory houses a variety of conventional machines used for determining the properties of both specimens and springs, covering the range of material diameters 0·004—2·0 in. These include a 30 ton universal hydraulic testing machine with seven load ranges—down to 0·3 ton—and equipment for autographic stress-strain curve recording. One machine of special interest is capable of developing a maximum torque of 120,000 lb. in., and is used to investigate the effects of hardenability on the static torsional properties of large diameter spring steel bars. An important part of the work in this laboratory is concerned with the correlation of tensile and torsional properties of existing and new materials for springs. The determination of the fatigue characteristics of drawn wires of diameters 0·01—0·25 in. is provided for by high-speed rotating-beam fatigue machines, which can complete up to 100 million cycles in as little time as one week.

Among the factors affecting the fatigue strength of springs made of various materials are the metallurgical structure and the surface condition. The structure can be changed by heat treatment and for this purpose furnaces operating at temperatures up to 1,400° C. are housed in a ground floor laboratory. They include units provided with facilities for treatment in a controlled atmosphere and others with air circulation equipment for operation at the lower end of the temperature range referred to above.

The fatigue life of spring materials can be considerably increased by surface treatments which induce compressive stresses in the surfaces. Shot-peening is ideal for this purpose and a unit is available in which the object under treatment is automatically rotated while at the same time being traversed by the shot stream. Research is in progress to determine the best conditions for peening and to compare the merits of various types of shot.

The study of the corrosion and protection of spring materials and the effects of hydrogen embrittlement due to electroplating is being continued in a new laboratory specially fitted out for this purpose. Equipment for electropating with copper, zinc, tin, cadmium and nickel has been presented by W. Canning & Co., Ltd., of Birmingham.

The laboratories on the second floor house machines for determining macro-hardness, tensile and torsional properties of wires, and load-deflection characteristics of small springs. Metallographic facilities are provided in specially equipped rooms for rough sample preparation, fine polishing and etching, microscopical examination, and photography. Equipment for determining the thickness of electroplated coatings is also available.

The Research Programme

The research programme is drafted with the object of solving major problems facing the industry, developing new materials and methods, and enabling the industrial products to perform satisfactorily under ever increasingly arduous conditions. The Research Committee examines proposals from member firms and from this compiles a short list of actual research projects. Reference is made below to a selection of investigations in progress or projected.

At one time springs were made only from carbon steel, a material possessing the prime requirement of high tensile strength. Over the years alloy steels have become increasingly important, and there is now an appreciable use of non-ferrous springs in special applications. Nevertheless, steel remains the most important spring-making material. Not unnaturally, therefore, many of the items in the research programme concern steel and steel springs. One of these—a study of the effect of variation in materials, methods of manufacture, and surface treatments on the fatigue properties of heavy suspension

springs—is part of a programme of work sponsored by the Ministry of Supply (F.V.R.D.E.). The object of another research project is to determine the maximum permissible section in order to obtain through hardening for various spring steels, and the effect of the percentage of martensite in the structure on the static and dynamic torsional properties of the steels. During the course of a study of the torsional fatigue properties of various steel wires, a comparison will be made between wires made from normal steels and others made from vacuum arc melted material.

The atmosphere in which a spring has to operate can be most important, as corrosion can accelerate failure. In corrosive atmospheres, such as in a chemical plant, the spring must therefore be protected, either by metallic or non-metallic coatings. This is not necessarily a straightforward solution as it can lead to other problems. As mentioned earlier, one of these is hydrogen embrittlement due to electroplating, in which hydrogen produced during the cleaning and plating processes actually enters the steel and causes premature failure Work is continuing in an effort to establish the optimum methods of hydrogen removal and to develop plating techniques which will prevent hydrogen embrittlement taking place.

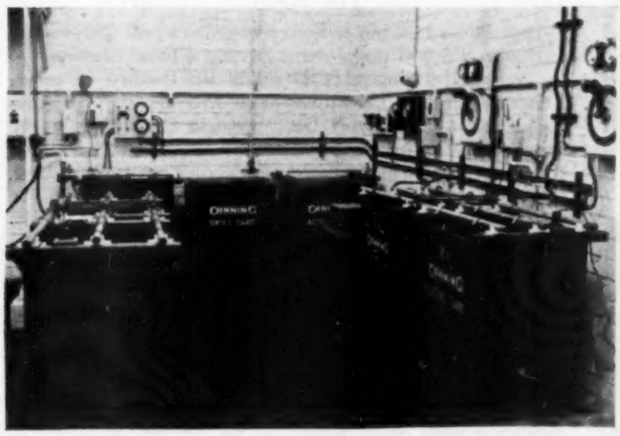
Another method of applying metallic coatings is peen plating. This is a trade name for a process in which the article to be coated is tumbled with glass balls and metallic powder while immersed in an appropriate liquid. The rubbing action of the glass balls has the effect of cold welding the powder to the article. One advantage claimed is the elimination of hydrogen embrittlement. The original processing technique (with zinc) resulted in a lowering of the fatigue limit in air, as compared with polished specimens, although no further lowering took place during corrosion fatigue tests. The coating produced by a revised technique was more adherent, but a high degree of porosity resulted in poor protection against a corrosive environment. There was no lowering of the fatigue limit when tested in air, as might happen with electroplating, neither was there any beneficial compressive stress as there is with zinc electroplating.

In the field of non-metallic coatings, the C.S.F.R.O. is exploring the application of new resinous and plastic materials to springs through an extra-mural project being carried out in the Engineering Department of Imperial College, London. Work to date includes the determination of the tensile strength, hardness, and abrasion resistance of the coating materials, and a preliminary series of corrosion fatigue tests. A fatigue test programme has now been drawn up which will include torsion and rotating bending tests on both polished and shot peened test pieces with the various coatings applied to them.

Most of the available information on the increase in the apparent elastic limit attainable by pre-stressing is limited to American materials, and to enable engineers in this country to take full advantage of this property when designing coil springs, curves relating wire diameter to the pre-stressed elastic limit are being constructed. They will indicate the maximum torsional solid stress to which a helical compression spring of given diameter and material may be designed. The effects of low temperature heat treatment on the above relationship is also being investigated.

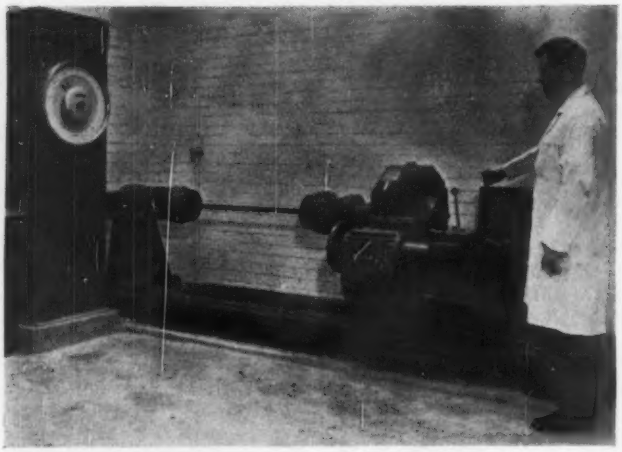
Low temperature heat treatment has also been studied in relation to the mechanical properties of 65/35 hard drawn brass spring wire. Besides raising the elastic properties, low temperature heat treatment also tended to stabilise the static tensile and torsional properties of conventionally drawn brass wire—an important factor when coil springs having consistent properties are required. It was found that low temperature heat treated material had an elastic limit in torsion approximately equal to 45% of the ultimate tensile strength.

The ever-increasing demands for higher operating temperatures for coil springs has revealed a dearth of information on the stress relaxation properties of the carbon and low alloy spring steels used in the United Kingdom. A programme of work has been instigated to



The electroplating laboratory for research into hydrogen embrittlement of high tensile steels, equipped by W. Canning & Co., Ltd., of Birmingham.

Loads of up to one ton are applied by this machine in order to study the load/deflection characteristics of heavy springs.



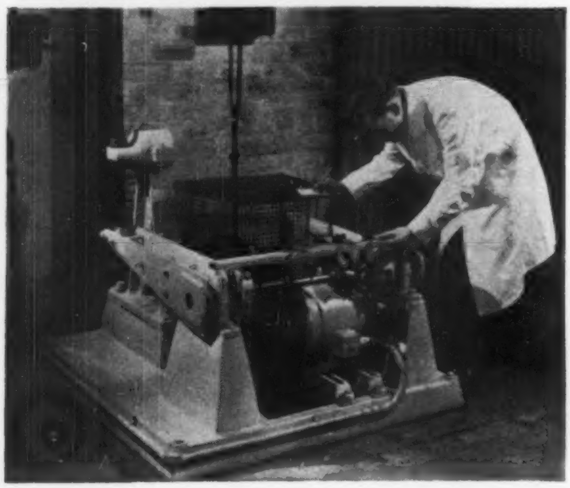
A machine capable of applying a torque of 120,000 lb.in. for examining the elastic and plastic torsional properties of large diameter hardened and tempered steel bars.

Determination of the torsional properties of fine gauge high tensile wires.

assess materials currently available, and ultimately to provide springs which will operate satisfactorily at temperatures as high as 850° C.

Titanium has a high corrosion resistance, high tensile strength, and low density, and it could be much more widely used for springs it if had a higher modulus of elasticity. Alloying additions can help in this respect, and a good deal of research has already been done on alloys of titanium in wire form : actual springs made of titanium alloys are now being studied. Expensive though titanium is, it has already found regular application as a spring material where corrosion fatigue is a serious problem and production loss due to replacement is an expensive item.

Copper-beryllium alloys, suitably heat treated, possess exceptional mechanical properties which make them useful spring materials. Inefficient solution treatment, however, may result in the formation of grain boundary precipitates, which, in turn, cause inconsistency in the response to ageing and unreliable mechanical properties. The extra-mural research at Sheffield University has as its object the understanding of the mechanism and effects of these grain boundary precipitates. Isothermal transformation studies have established that their rate of formation is influenced by such factors as composition, grain size and cold working. In order that the results of this fundamental work may be applied to production processing, a complementary study on the dynamic and static properties of appropriately treated copper-beryllium has been undertaken at the C.S.F.R.O. headquarters.



Torsional fatigue testing of hardened and tempered cylindrical specimens.

The Future

The spring-making industry is quite small, employing only about 7,000 people and having a total turnover in the region of £5m. It is made up mainly of small companies, most of which have less than a hundred people on the pay-roll but, because the spring is so universally employed, research is essential. The C.S.F.R.O. has a total membership of nearly a hundred, of which only half are spring makers. The rest are manufacturers in the supplier or user industries—such as wire producers or machine tool makers—who are glad to support an organisation which aims at giving them better components. More of these associate members—as the non-spring-makers are called—are likely to join the Organisation as the new research programme gets under way.

The Organisation receives a grant of £4,300 a year from the Department of Scientific and Industrial Research and, as it grows in the future, it hopes to attain full research association status so that it may take its place among over forty other industrial research associations in the Government scheme.

Old Zirconia and New Zirconium

By M. Schofield, M.A., B.Sc., F.R.I.C.

With the new emphasis on zirconium of high purity for the atomic energy industry, it is absorbing to follow the years of endeavour which involved reduction techniques of a high order. Zirconium owes its discovery to the analytical skill of Klaproth towards the end of the eighteenth century, but it is only in comparatively recent years that it has become a metal of engineering importance.

THE present emphasis on zirconium as an invaluable metal in nuclear engineering for fuel canning or sheathing illustrates the striking contrast between a relatively new metal and its minerals known to the ancients. Zirconium of high purity is less than fifty years old, being first produced when Lely and Hamburger, of the Philips Lamp Works at Eindhoven, used a " bomb " process in which pure sodium reacted with re-sublimed zirconium chloride. In contrast is a Biblical reference to " jacinth " or " hyancinth " among precious stones. Zircon was used in ancient times for intaglio work, while centuries ago the mineral with the peculiar name of hyancinth (an orange-red zircon) was rivalled by a second zirconium mineral " jargon," the name given to colourless or yellowish varieties of the same mineral. While the earth zirconia lost industrial applications when the Welsbach gas mantle and Nernst lamp declined, this was more than compensated for when zirconia crucibles, muffles and high-grade refractories, withstanding temperatures up to 2,300° C. and of high mechanical strength, took an unrivalled place for use in electric furnaces and in refining precious metals, this apart from an increasing demand in electrical porcelains such as sparking plugs, and in zirconia vitreous enamels, where zircon " flour " became an opacifier.

Work of Klaproth

Not until the last decade or so of the eighteenth century was an unknown element or metal detected in zirconia, this when Klaproth, Germany's most brilliant analyst of his day, outwitted such French chemists as Vauquelin and Guyton-Morveau. It seems a coincidence that uranium and zirconium should be linked in this atomic age, for these two elements owe their discovery to the analytical skill of Martin Heinrich Klaproth. Metallurgy owes much to this German chemist. His name crops up again and again, in the history of tellurium and cerium, and in naming titanium (from " Titans, the first sons of the earth," as Klaproth insisted, rather than use " Menachanite," derived from the parish of Menachan in Cornwall where parson Gregor first stumbled on a black titanium mineral). Even beryllium, that other " atomic " sister element to zirconium in reactors, was so named when Klaproth disdained such a sugary name as " glucinum." After a first class training in Wendland's Berlin pharmacy " at the Sign of the Golden Angel in the Street of the Moors," Klaproth took over Valentine Rose's chemical work, with Rose junior as assistant to check all Klaproth's analyses. Klaproth was especially interested in gem-stones, an interest bringing fruitful results in the chemistry of metals. It was in analysing a zircon specimen from Ceylon that he discovered a new earth, his " Zirconerde " or ZrO_2 composing 70% of the total, though Bergman had missed zirconium entirely in days when confusion with alumina was common. There were a number of elements present at times so that it is not surprising that such confusion existed, that only an exceptional analyst of the period met with success. There had been claims for another earth in zircons, this being rather optimistically named " noria " and signifying a new element " norium " which was not to appear at the Chemist's Table. Later, in 1869, Sorby in the *Chemical News* announced a new element " jargonium " in zircon or Ceylon jargon, yet this was due to small proportions of uranium present. Then Church in the *Chemical News* described his work on the spectrum of zirconium and uranium, and suggested that a new element " nigrium " was present. Even as late as 1901, in the *Berichte*, there was a claim by Hofmann and Prandt for a new earth " euxenerde." Such claims bring to mind less conscientious ones made at the time when Welsbach, after trying zirconia for his gas-mantle research, patented his " Auer Mixture " of rare earths, others then seeking to avoid having to use his patent by specifying new elements like " lucium," " kosmium " and " neokosmium."

Liberation of the Metal

Early last century there came attempts to reduce zirconium compounds to liberate the metal present in zirconia. The highly inert oxide proved of no use as raw material, this when Davy attempted to decompose it, while others had struggled with attempts to reduce it in hydrogen (which would have formed the hydride in any case.) Berzelius was the first to succeed with his favourite technique, using potassium metal in layers alternating with the double fluoride of zirconium and potassium in a small iron tube within a platinum crucible. Amorphous zirconium metal with zirconia was the product when the residual salt dissolved in water, the black powder being described as resembling charcoal. Digestion of the residue with dilute acid and washing with ammonium chloride and then with alcohol were also used in this first reduction process, yet the product was so impure that Berzelius could not convert it into anything resembling a metal by compressing or polishing. This double fluoride of zirconium and potassium proved a useful raw material, with aluminium later used as reducing agent, although alloying with this metal vitiated attempts to obtain purer zirconium by reduction in a graphite crucible. Before sodium and magnesium reduction processes assumed major importance, the use of aluminium was persisted with even in aluminothermy, when a " difficult " slag of zirconia and alumina negatived this idea. Even in reduction of the double fluorides with aluminium using a large excess, it was found that a

maximum purity of 99·5% zirconium was the best obtainable, this after removing the large residue of aluminium.

In the preparation of "raw" or impure zirconium for subsequent purification by the iodide or other refining process, calcium reduction of zirconia was shown possible. Calcium was studied for some years from 1902 by Wedekind in Germany, while De Boer and Fast also tried calcium as alternative to sodium, finding that excess calcium gave fine zirconium particles leached from the melt. Kroll also tried out such calcium reduction, this time in an argon or inert atmosphere and using a calcium chloride flux to give zirconium of 99·5% purity. Yet calcium did not survive when magnesium and sodium reduction processes became widely studied. Interest in aluminium revived for a time with an arc process working *in vacuo* for volatilizing aluminium, rods of alloy containing up to 27·5% aluminium being used. Marden and Rich improved on such processes in 1921 by suggesting hydrogen for sweeping out gases, then evacuating the container, and using an arc to yield 99·6% purity zirconium. While summarising the varied techniques which failed in the face of reduction by magnesium or sodium, mention may be made of the failure of attempts to use electrolytic methods for zirconium. Becquerel in 1832 tried in vain to prepare the metal by electrolysis of zirconyl chloride solution, while later Troost experimented with an electrolysis of the molten double fluoride and claimed the production of lustrous laminae. An American concern, namely Horizons Incorporated of Cleveland, Ohio, also gave considerable attention to the problem of producing zirconium from molten salt baths, but the method has not been adopted on the commercial scale.

Sodium and Magnesium Reduction

In place of the Berzelius reduction process using potassium and a zirconium halide, a sodium reduction proved most promising until magnesium became even more favoured. Berzelius, like others who failed to make the ductile metal, was bedevilled by the oxide, due to the high affinity of zirconium for oxygen, even at moderate temperatures. Later, the property of absorbing oxygen and nitrogen brought the use of zirconium as a "getter" for removing the last traces of gases in vacuum tubes. As early as 1865 Troost, in a paper on "Recherches sur le Zirconium" published in Paris, tried sodium in place of potassium, this before he also experimented with magnesium as reducing agent. Further efforts to reduce zirconium chloride vapour with sodium followed, a reduction foreshadowing the new production of "hyper-pure" silicon by reaction of pure $SiCl_4$ with sodium. In addition to the use of sodium by Lely and Hamburger already referred to, there were other workers in the United States such as Hunter and Jones and also Cooper, who in 1923 described their studies of sodium reduction in the *Transactions of the Electrochemical Society*. W. J. Kroll, noted for his production of titanium, discussed in the *Journal of the Electrochemical Society* in 1950 the relative merits of sodium and magnesium for reduction, and also pointed out that the use of the mixed metals would be of some advantage in giving as end-product a low-melting salt less hygroscopic than magnesium chloride. One disadvantage of sodium is the larger mass of metal and of salt formed in the reaction, while the higher volatility of sodium is also undesirable.

For production of zirconium from its tetrachloride, the use of magnesium as reducing agent has taken precedence for large-scale work and for yielding zirconium of the required purity. Von Zeppelin, in patents in Germany and the United States in 1940, took to magnesium in place of calcium or sodium, the zirconium liberated being in the form of a powder contaminated with oxygen as well as magnesium. The reactions with both zirconium chloride and the double chloride of zirconium and sodium were studied, and although zirconium settled below the melt, there was difficulty with the pyrophoric nature of the metal. Since zirconium chloride, in contrast to titanium chloride, is a solid with a high affinity for moisture, addition to molten magnesium will yield a metal with oxygen contamination from the oxychloride; hence the chloride is vaporised in a separate vessel to leave behind the oxy-compounds, the vaporising in a hydrogen atmosphere assisting in avoiding iron impurities. Kroll's technique brought to high efficiency at the U.S. Bureau of Mines, Albany, differed from the well-established process for titanium, in that the metal sponge left from reaction of metal chloride with magnesium did not prove amenable to the leaching method for removing magnesium salts plus excess metal. Hence, in the case of zirconium, a vacuum distillation process was adopted for removing such residues, the zirconium sponge remaining being then briquetted in a press and converted in an arc furnace to zirconium ingots.

The Iodide Process

To complete the picture of the metallurgists' struggles to produce an "obstinate" metal in a high state of purity, the "Iodide Process" may be mentioned as a modern chapter in the zirconium story begun by Berzelius. Van Arkel, in his classic papers in the *Zeitschrift fur Anorganische Chemie* for 1925, began this technique of decomposing a metal halide vapour on a hot filament, titanium, hafnium and thorium being metals thus prepared in a high state of purity. Since De Boer and Fast also used this idea, the name "Iodide Process" was adopted for a process in which those traces of oxygen and nitrogen hitherto vitiating the metal produced have been eliminated. At first a hot tungsten filament was used for decomposing ZrI_4 in an evacuated vessel, this being improved by a pure zirconium filament to avoid alloying with tungsten. Iodine within the vessel reacts with crude zirconium, the iodide diffuses to the hot filament, and zirconium metal is deposited by keeping the filament at temperatures round about 1,250° C. The Foote Mineral Company, the Battelle Institute, and the Westinghouse Company have so extended this iodide process as to produce zirconium bars of 12 ft. in length. This "iodide zirconium" represented the ultimate high standard of purity; yet with atomic energy commissions accepting other hafnium-free standards of zirconium produced by Carborundum Metals Company and by I.C.I. in this country, vacuum processing is now used to avoid the oxygen and nitrogen impurities.

Applications

Zirconium metal has some striking properties which have given rise to potential and practical applications. While chemical engineers saw much promise in its

(*continued on page* 166)

The Effect of Internal Oxidation on Some Properties of Copper Alloy Sintered Compacts

By R. H. Seebohm and J. W. Martin

Department of Metallurgy, University of Oxford

Beryllia and alumina dispersions have been produced in copper powder compacted specimens by internal oxidation of suitable alloy powders. Density, hardness and compression yield-points have been determined for the alloys after different sintering treatments, and dispersion hardening has been observed in the internally oxidised compacts. The structures have been examined metallographically, and both optical micrographs and electron micrographs of carbon extraction replicas from internally oxidised compacts are presented and discussed.

A NUMBER of workers have investigated the use of internal oxidation to obtain dispersion hardening[1-4]. In this technique, an alloy consisting of a dilute solid solution of a base metal in a more noble metal is heated in oxidising conditions, so that oxygen diffuses into the alloy. When the conditions are suitable, a fine dispersion of the base metal oxide is formed in a matrix of the noble metal. These oxide dispersions are stable at temperatures up to that of their formation, which can be close to the melting point of the alloy.

Published work in this field has so far been confined to solid alloy specimens. The time required for the complete internal oxidation of large cross-sections is very long, due to the parabolic decline in oxygen diffusion rate as the distance from the surface is increased. Many hours are required to oxidise to a depth greater than one or two millimetres. Furthermore, concentration gradients are set up as internal oxidation proceeds, so that the dispersions are not uniform throughout a specimen.

Experiments have therefore been started at Oxford using powder alloys. With copper alloys of an average granule size of 30 microns, internal oxidation is complete in 30 seconds or less at 750° C., or above. It is clearly convenient to combine the oxidising heat treatment with the sintering operation, following cold pressing. This can be done by using as a source of oxygen a scale of copper oxide on each granule, formed by heating the loose powder in air.

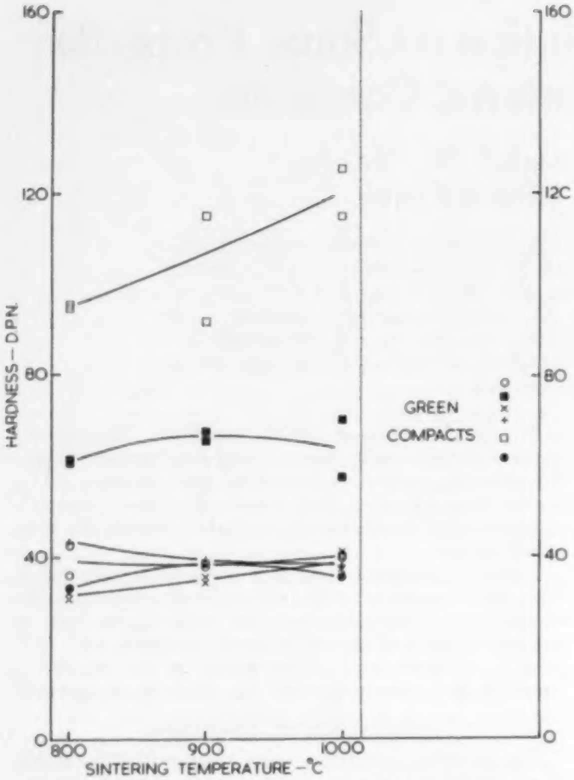
Internally oxidised compacts have been prepared in this way, together with un-oxidised specimens for comparison. The measurements made have been of density, Vickers diamond pyramid hardness and yield point in compression. Observation of the structures obtained has been attempted by electron microscopy.

Preparation of Specimens

The powders available were copper–0·19% aluminium, copper–0·38% beryllium, and unalloyed copper; all atomised powders of particle size −200 mesh. They were pressed in a cylindrical die to produce compacts about 1 cm. in diameter by 1 cm. high: a die 0·7 cm. in diameter was used for some later experiments. The compacts to be internally oxidised, made from powder heated in air at 200° C. or 300° C. until they had gained the weight of oxygen required, were sintered in argon.

TABLE I.—DENSITIES OF COPPER ALLOY POWDER SPECIMENS

		Internally Oxidised			Vacuum Sintered (not oxidised)			Green Compacts	
	Compacting Pressure (tons/sq. in.)	Sintered at 1,000° C.	Sintered at 900° C.	Sintered at 800° C.	Sintered at 1,000° C.	Sintered at 900° C.	Sintered at 800° C.	With oxide scale	Without oxide scale
Copper–0·38% Beryllium	30	7·40	7·26	7·20	7·78	7·72	7·77	7·34	7·90
		7·34	7·15	7·15	7·99	7·67	7·80		
	22½	6·98	6·88	6·78				6·97	
		6·97	6·86	6·87					
	13½	6·31	6·16	6·22				6·26	
		6·26	6·22	6·21					
Copper–0·19% Aluminium	30	7·57	7·52	7·45	7·75	7·48	7·41	7·61	7·90
		7·55	7·54	7·29	7·75	7·52	7·36		
	22½	7·29	7·18	7·22				7·17	
		7·27	7·19	7·16					
	13½	6·57	6·54	6·51				6·50	
		6·64	6·52	6·49					
Copper	30	8·01	7·87	7·83	8·06	7·99	8·07	7·77	7·85
	22½	7·66	7·50	8·04				7·43	
	13½	7·28	7·10	6·92				6·78	



Open squares	0.38% Be-Cu (oxidised)
Solid squares	0.19% Al-Cu (oxidised)
Open circles	0.38% Be-Cu
Solid circles	Copper with oxide scale
Upright crosses	Copper
Diagonal crosses	0.19% Al-Cu

Fig. 1.—Vickers diamond pyramid hardness values of powder alloys showing effect of internal oxidation. Compacting pressure : 30 tons/sq. in.; sintering time : 2 hours.

The unoxidised compacts were sintered in vacuum, the sintering time being 2 hours in all cases. The compacting pressures used were 13½, 22½ and 30 tons/sq. in., and the sintering temperatures 800° C., 900° C. and 1,000° C. The effects of these variables on density are shown in Table I.

Mechanical Properties

The Vickers diamond pyramid hardness numbers were measured using a 10 kg. load, and the yield points were determined from compression tests carried out on a Hounsfield Tensometer. The results of these tests are shown in Figs. 1 and 2. These values are taken from the specimens pressed at 30 tons/sq. in.; the mechanical properties of those compacted at lower pressures are impaired by lack of homogeneity.

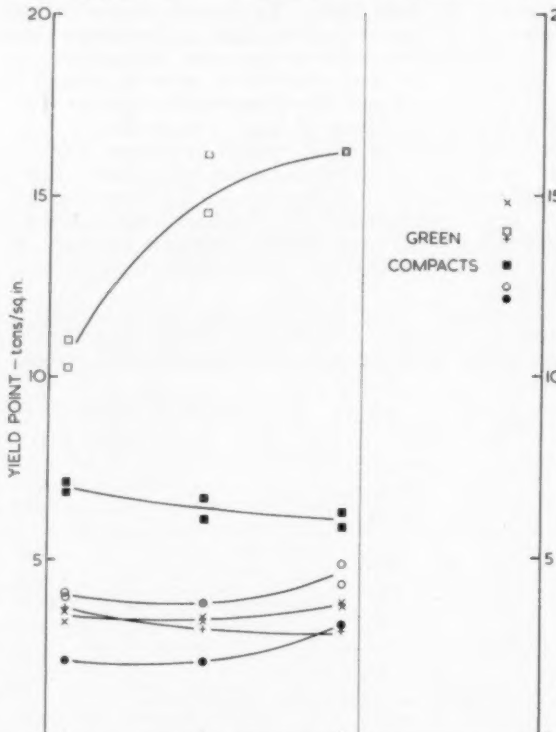
The specimens prepared so far have been rather brittle, although some plastic deformation can be achieved. It has not yet been possible to measure their ductility, as the compacts are too small to machine tensile specimens from them.

Metallography

Solid copper-aluminium and copper-beryllium alloys, when internally oxidised, produce particles which are very fine close to the surface and become coarser with increasing depth. For example, at about 0·5 mm. from the surface of a solid specimen of copper–0·34% beryllium, internally oxidised at 950° C., the particles of BeO, as measured from electron micrographs, are about 5,000Å in diameter. At 0·1 mm. from the surface, however, they are about 500 Å.

Since the average diameters of the powder granules used in the work described is about 0·03 mm., the oxide particles may be expected to be very fine indeed. In fact in electron microscope examination of carbon extraction replicas from powder compact surfaces, oxide particles have not usually been identified (Figs. 3–6). Only one replica (Fig. 6) showed particles, which may have formed towards the centre of an exceptionally large granule.

Solid internally oxidised materials usually have large quantities of oxide in the grain boundaries, giving rise to intergranular brittleness [3, 5, 6]. As far as can be seen, internally oxidised powder compacts, when broken up by compression, tend to fail along the boundaries of the original powder granules (Fig. 7). Fig. 5 shows oxide in grain boundaries, which is probably not continuous enough to lead to weakness, and so the efficiency of the sintering process itself may therefore be questioned.



Open squares	0.38% Be-Cu (oxidised)
Solid squares	0.19% Al-Cu (oxidised)
Open circles	0.38% Be-Cu
Solid circles	Copper with oxide scale
Upright crosses	Copper
Diagonal crosses	0.19% Al-Cu

Fig. 2.—Compression test yield points of copper powder alloys showing effect of internal oxidation. Compacting pressure : 30 tons/sq. in.; sintering time : 2 hours.

Fig. 3.—Electron micrograph of shadowed carbon extraction replica of copper–0·19% aluminium powder compact, sintered and internally oxidised at 1,000°C. Etched ferric chloride. ×1,600

Fig. 4.—Electron micrograph of shadowed carbon extraction replica of copper–0·38% beryllium powder compact, sintered and internally oxidised at 800° C. Etched ferric chloride. ×1,800

Fig. 5.—Electron micrograph of shadowed carbon extraction replica of copper–0·38% beryllium powder compact, sintered and internally oxidised at 1,000° C. Etched ferric chloride. ×16,000

Fig. 6.—Electron micrograph of unshadowed carbon extraction replica of copper–0·38% beryllium powder compact, sintered and internally oxidised at 1,000° C. Etched ferric chloride. ×21,000

Fig. 7.—Optical micrograph of section across fracture surface of copper–0·38% beryllium powder compact, sintered and internally oxidised at 1,000° C. Edge protected by electrodeposited copper. Etched ferric chloride. ×300

Discussion

It can be seen that the mechanical properties of powder compacts can be altered considerably by internal oxidation. It is also to be expected that their creep resistance, in particular, may be improved. This has been demonstrated in the case of solid oxidised alloys[7, 8], although Catterall and Jenkins[9] find that the high temperature tensile strength falls off more rapidly than might be expected with increasing temperature of testing. It is hoped that high temperature tensile testing of powder compacts will be possible as a continuation of the present work.

With regard to the structure shown in Fig. 7, a factor that might cause weakness between the powder granules is the film of copper oxide there (containing in addition some of the base metal oxide), which is used as a source of oxygen. Hoar and Butler,[10] working with electrolytic powders, have found that oxide films are tenacious, and reduce green density most at about 1% by weight of oxide, which is comparable to that used here. They observed the films to break up and cause shrinkage on sintering.

In the case of internally oxidising alloys, the oxygen is removed leaving reduced copper. If this is in fact formed as a film surrounding each granule, its thickness has been calculated to be about 1,000 Å for the copper–0·38% beryllium alloy. No such films have been observed in electron microscope replicas, although there are areas which may well be solid lumps of reduced copper (Figs. 3 and 4).

It is hoped in later experiments to investigate the effect of supplying oxygen from powdered metal oxide physically mixed with the alloy powder. (This may, incidentally, enable the amount of oxygen available to be controlled more closely).

Experiments have been started on nickel-based alloys, containing aluminium and beryllium, which are likely to be of greater technological value than copper alloys. Preliminary work is well in hand, and the results will be published in due course.

Acknowledgment

The authors are grateful to Professor W. Hume-Rothery, O.B.E., F.R.S., Isaac Wolfson Professor of Metallurgy at Oxford, for his encouragement, and for the laboratory facilities made available.

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Old Zirconia and New Zirconium

(*continued from page* 162)

exceptional corrosion-resisting character, with zirconium claimed superior to any other metal in rayon spinnerets, this promise has not resulted in large-scale adoption in place of tantalum (or platinum-gold in the case of spinnerets). In contrast, this corrosion-resistance combined with high mechanical strength has enhanced its value in the atomic energy field, where its low neutron absorption and compatibility with uranium have brought wide application. For fuel sheathing and other components, zirconium ingots or alloyed zirconium ingots of up to 3 tons are arc-melted, forged, extruded or rolled, with full attention to avoid contamination by nitrogen. Apart from such special applications, the progress of the metal first liberated by Berzelius will provide an absorbing study. In smokeless flashlight powders, in ammunition primers, in "getters" for many types of radio valves and in other fields, the metal is certainly as bright as those zirconium fires which no water, carbon dioxide, foam or carbon tetrachloride can extinguish !

Trancoa Introduces Silicon Rods

New Metals and Chemicals, Ltd., of Chancery House, Chancery Lane, London, W.C.2, sole U.K. distributors of hyper-pure silicon produced by Trancoa Chemical Corporation, have announced the availability of polycrystalline silicon rods for floating zone crystal growing. The Trancoa rods are claimed to be unique in that they are centreless ground from silicon that has not been previously melted. As a result, the rods are very uniform in diameter, and have a very low boron content and a density very near theoretical limits. These characteristics result in a greater ease of producing single crystals by the floating zone method. The rods are offered in standard diameters of $\frac{3}{4}$ and $\frac{7}{8}$ in. with tolerances of plus or minus 0·005 in. Other diameters from $\frac{3}{8}$ to 1 in. are available on a made-to-order basis at no increase in price. The nominal length of the rods is 10 in. and they have a boron content of approximately 1 in 10^9 parts.

Surface Conformation of Metals Under High Nominal Contact Pressures

By L. H. Butler, M.Sc.(Eng.), M.Sc.(Wales), A.M.I.Mech.E., A.F.R.Ae.S.

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The surfaces of materials produced during processes involving bulk plastic deformation (wire drawing, strip rolling, impact extrusion, etc.) are frequently required by the consumer to have a bright polished appearance. This implies conformation between the deforming tool and the deformed material without metallic pick-up and scoring occurring, any relative motion giving a burnishing action. This contribution discusses with experimental evidence the factors, particularly lubricant characteristics and metal surface topography, which may affect the degree of conformation. It is shown that a thin, semi-continuous lubricant film may easily be mechanically trapped at the tool to metal interface, the metal subsequently being deformed largely via fluid pressure. Such conditions lead to the production of a matt, non-reflective surface on the deformed material. It is suggested that such conditions are not infrequently encountered in practical forming processes and that they contribute in part to the relatively low coefficients of friction sometimes quoted for these processes.

IT is accepted that on a microscopic scale few engineering surfaces can be considered flat. They usually contain random undulations (asperities) on which is sometimes superimposed a well defined and orientated roughness, characteristic of the surface production process. Several methods exist for assessing surface texture, including stylus tracing, taper sectioning, comparison microscopy, and optical interference. Probably the most commonly used method is that of stylus tracing, in which a stylus with a tip radius of 0·0001 in. or less is drawn under light load across the surface. A trace is made automatically in which, since the crest to trough heights of the asperities are usually so small (being measured in micro-inches), a considerably distorted picture must for convenience be presented. The trace is magnified in both the horizontal (surface plane) and vertical (roughness) directions. Vertical magnifications ranging from × 1,000 to × 50,000 and horizontal magnifications of × 100 and × 500 are commonly used. Depending on the ratio of vertical to horizontal magnification chosen, therefore, an exaggerated impression of the surface roughness is conveyed by cursory inspection of the trace. The same impression is also conveyed by the interference and taper sectioning methods.

Whilst it is generally appreciated that the trace as presented is distorted, the degree of distortion is frequently underestimated, and it is only by replotting the traces on the realistic basis of unity ratio of vertical to horizontal magnification that a true appreciation of the surface profile is obtained. R. E. Reason[1] makes this point in stating that rarely are the slopes of the asperity flanks inclined at more than 5° to the general surface plane.

This fact is of importance when considering metallic contact, particularly under pressures sufficient to cause bulk plastic deformation, such as occur in any metal forming process. The impression gained by casual interpretation of the surface trace, that the asperity tips will be bent over and that progressive plastic deformation of the tips on initial loading will lead to the incorporation of minute surface folds, is not necessarily correct, and it is more likely that the progressive deformation will result in the elevation of the troughs until perfect or near-perfect, conformation of the deforming tool or die and the deformed metal occurs in the presence of surfaces where no thick contaminant or lubricant film is present.

When two bodies are in contact they are normally in the state of bulk elastic contact, i.e. the only plastic deformation occurring is localised at the minute contacting areas of a few asperities. Any load between the bodies is supported at these discrete points, where the true contact stress usually exceeds the yield stress of one or both bodies. The local deformation may cause breakdown of contaminant or lubricant films, resulting in metallic adhesion or welding which, Bowden[2] suggests, is the primary cause of sliding friction.

If, however, the load between the bodies is progressively increased until such time as bulk yielding of one of the bodies occurs (i.e. the state of bulk plastic contact), the interface contact conditions are inevitably modified, and consequently it might be assumed that the nature of the frictional resistance would in itself be modified. Conditions such as these are present in any metal forming process, and it is of interest to establish the conditions under which interface surface conformation will or will not occur during bulk plastic flow.

The author has previously suggested[3] that the presence or absence of a lubricant, the physical properties of the lubricant used and the topography of the contacting surfaces are the primary factors affecting the degree of surface conformation and, with reservations, the frictional resistance. In a series of experiments[4] in which soft aluminium discs were plastically deformed between flat steel dies with highly polished surfaces, it was shown that the surface finish of the discs after deformation varied from a mirror finish when using no lubricant or one of very low viscosity, progressively through to a dull non-reflective finish using a highly viscous lubricant. It was suggested that near perfect conformation of the die and specimen surfaces occurred in the first case, whereas with the heavier lubricants mechanical trapping of lubricant films between the surfaces caused deformation to occur largely via fluid pressure, as opposed to die-to-metal contact. In the absence of this contact no burnishing of the deformed surface could occur to give it a polished appearance.

To support the view that the presence and properties of the lubricant materially affect the degree of surface con-

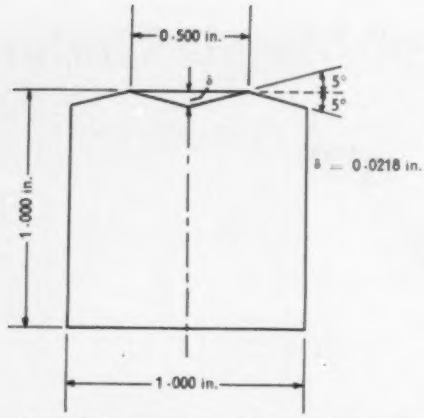


Fig. 1.—Nominal dimensions of coned steel specimens. (Diametrical section through specimen).

formation, further experiments have been carried out in an attempt to show clearly on a macroscopic scale the effects which it is thought also occur microscopically in practice.

Three series of compression tests were conducted in which the surfaces of either the die or the material to be deformed were artificially "roughened" on a macroscopic scale. Compression tests were then made with and without lubricants present at the die-to-metal interface, the deformed metal surfaces being subsequently inspected to determine the effect of the lubricant present on the die-to-metal surface conformation.

EXPERIMENTAL

A series of paraffinic base oils was used, the details of which are given in Table I. The lubricants contained no additives to improve their boundary or extreme pressure behaviour.

Compression of Coned Steel Cylinders between Flat Dies

Compression specimens of the nominal dimensions shown in Fig. 1 were made from bright drawn mild steel bar, the 5° cone flank angle being chosen as representative of the flank angle of a microscopic asperity. A tensile test on the material showed it to be in the work hardened condition with a high ratio (0·9) of yield to ultimate stress. Its modulus of elasticity was 30×10^6 lb./sq. in., U.T.S. 35 tons/sq. in., reduction of area measured on original area 58·4%, and Brinell hardness 173. The specimens were compressed between hardened steel dies with flat polished surfaces.

Initial tests were made by loading in 5 ton increments from zero to 45 tons with no lubricant present (the surfaces having been swabbed with a solvent), to determine the manner in which deformation progressively occurred. After each load increment each of the dimensions shown in Fig. 2 was measured. The gauge length l was scribed

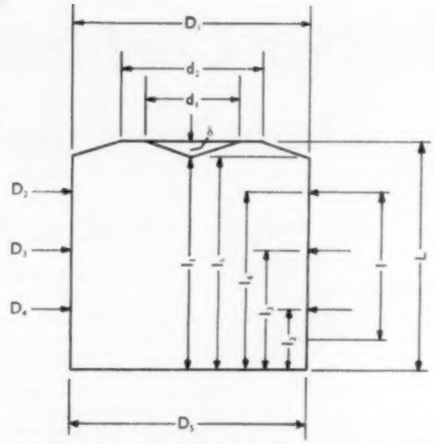


Fig. 2.—Relevant dimensions of coned steel specimens.

TABLE I.—VISCOSITY OF LUBRICANTS USED FOR TESTS

Lubricant Reference	Viscosity (centistokes at 100° F. and at atmospheric pressure).
1E	4·45
5B	31·8
12B	113·0
13B	173·9
19	820·0

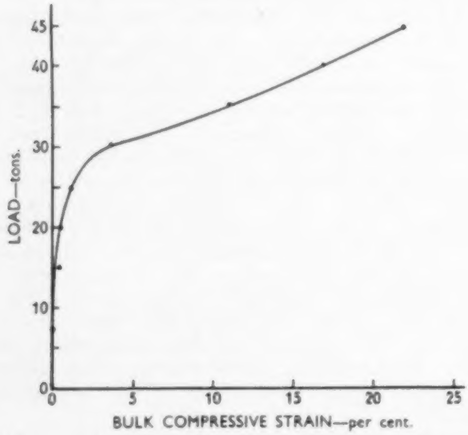


Fig. 3.—Bulk strain v. load. (Coned steel specimen).

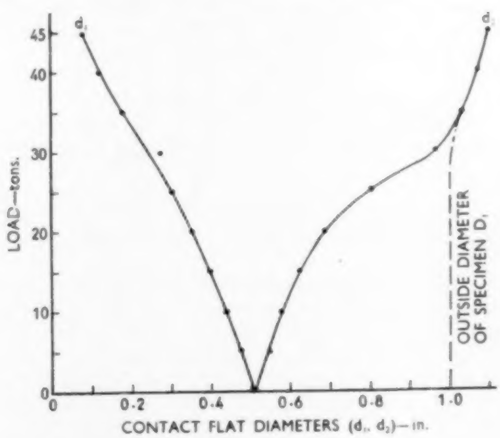


Fig. 4.—Change of contact flat diameters with load. (Coned steel specimen).

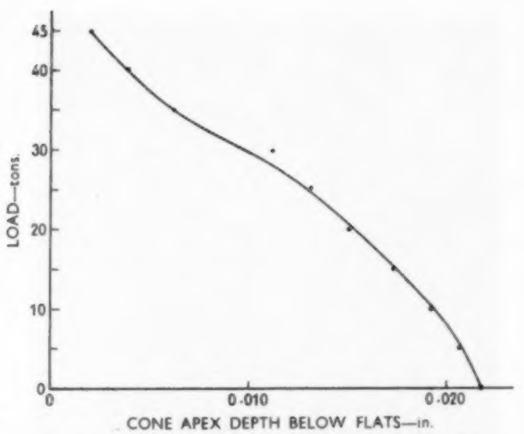


Fig. 5.—Cone apex depth v. load. (Coned steel specimen).

to enable an estimate of the compressive strain in the bulk of the specimen to be made as distinct from the localised deformation on the profiled face. As loading increased, the original ring contact changed to a flat annular contact area, and the re-entrant cone apex depth (δ) was measured from this face. The variation of the numerous dimensions with load is best seen by reference to the plotted figures.

Fig. 3 shows the bulk compressive strain of the specimen plotted against load. Only at loads exceeding 30 tons did this strain become appreciable, the greater part of the deformation prior to this being restricted to the profiled face of the specimen. Fig. 4 shows the change of the contact flat inner and outer diameters with load. It is seen that considerable conformation of the die-to-metal interface occurred before the load (30 tons) was reached at which bulk deformation was appreciable. At the same time the cone apex depth below the flats (Fig. 5) decreased rapidly, becoming approximately 50% of its original depth at 30 tons load. Finally a plot (not reproduced here) of the progressive change in the cylindrical

TABLE II.—DETAILS OF TEST CONDITIONS

Specimen Reference	Lubricant Reference	Loading	Lubricant Application
A	Nil	Direct to 35 tons	Nil
B	1E		Lubricant applied liberally to fill cone and cover surface
C	19		
D	13B		

shape of the specimen, shows that the initial lateral spread was confined mainly to the profiled end, and that only after die-to-metal conformation was well established did the normally expected symmetrical barrelling occur.

From the initial tests it was apparent that a direct load on such specimens to 35 tons to determine the effect on surface conformation of interposed lubricants would be suitable. It would give a measurable degree of bulk plastic deformation, and ensure that the outer contact flat diameter extended to the full specimen diameter and that a central deformed cone of measurable size remained. Four specimens were tested under the conditions set out in Table II.

After deformation the following dimensions, identified in Fig. 2, were taken on each specimen: d_1, d_2, δ, L, l, D_1 and D_5. The gauge length measurements showed that each had suffered approximately 12% bulk compressive strain, and that the outer contact flat diameters (d_2) coincided with the specimen diameters (D_1). The presence of the lubricants, however, showed a marked effect on the remaining cone impressions as shown in Fig. 6. Here the contact flat inside diameter (remaining cone diameter), and the cone apex depth below the contact flat are plotted against lubricant viscosity.

In the absence of a lubricant, near 100% conformation of die and metal occurred, the remaining cone diameter having decreased from 0·498 in. to 0·114 in., its apex depth meanwhile reducing from the original 0·0218 in. to 0·0025 in. The presence of even the lightest lubricant (1E), however, left a pronounced cone, whilst with the heavier lubricants the cone dimensions were little different from the original.

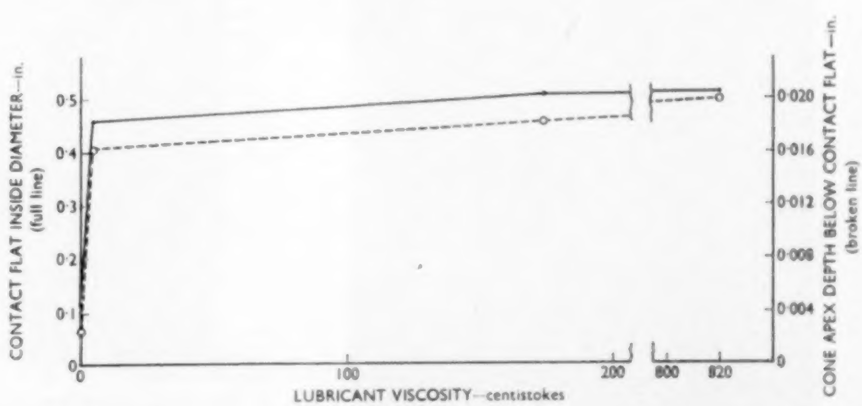
The photographs in Fig. 7 show clearly the difference between specimens A and C after loading.

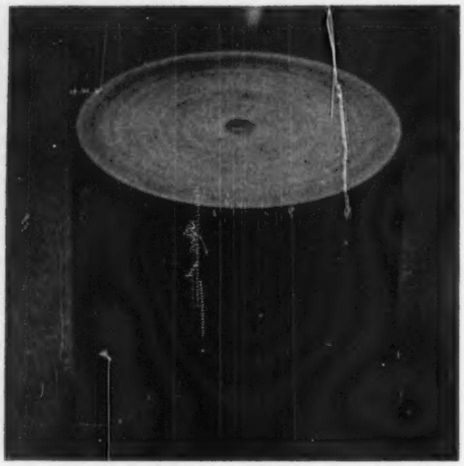
Compression of Aluminium Discs between Profiled Dies

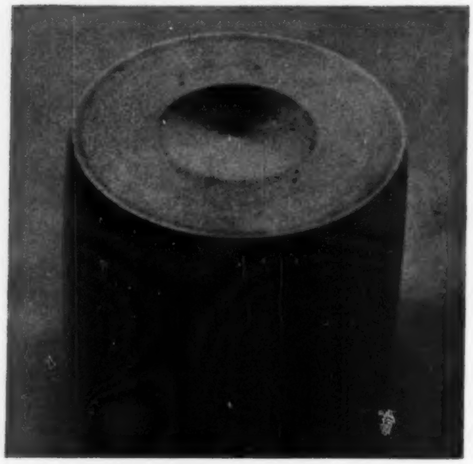
The compression specimens were made 0·625 in. diameter × 0·139 in. thick from Noral 2S hot mill strip conforming to B.S. 1470 S1C, of Brinell hardness 23·0. This corresponds almost to the fully annealed condition. The material has a nominal composition of 99½% aluminium with a typical U.T.S. of 5¼ tons/sq. in. and an elongation of 35% on 2 in. gauge length. Each specimen was surface polished to remove all evidence of rolling marks, leaving a flat although not necessarily highly polished surface.

Two profiled compression dies were made, their profiles being formed as follows :—

Fig. 6.—Effect of lubricants on remaining cone dimensions. (Coned steel specimens: load 35 tons).

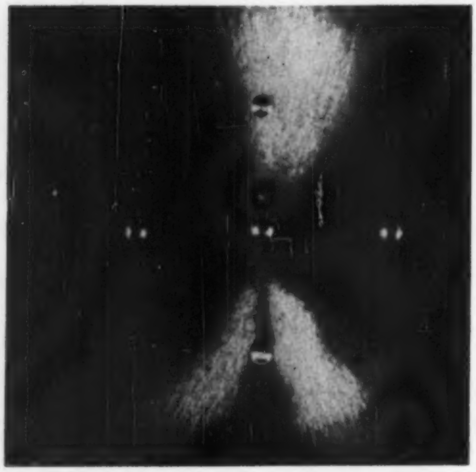






Specimen A **Specimen C**

Fig. 7.—Coned steel specimens after deformation. × 2 approx.



Impression	Depth (in.)
Top	0.0029
Bottom	0·0026
Right	0·0036
Left	0·0037
Centre	0·0035

Fig. 8.—Multiple impression die and details. × 4 approx.

TABLE III.—TEST CONDITIONS WITH MULTIPLE IMPRESSION DIE

Specimen Reference	Lubricant Reference	Lubricant Application
1m	Nil	Nil
2m	1E	Thin smear to specimen surface
3m	1E	Applied to die surface in sufficient quantity to ensure filling of impressions and covering of die surface
4m	5B	
5m	12B	
6m	13B	
7m	19	

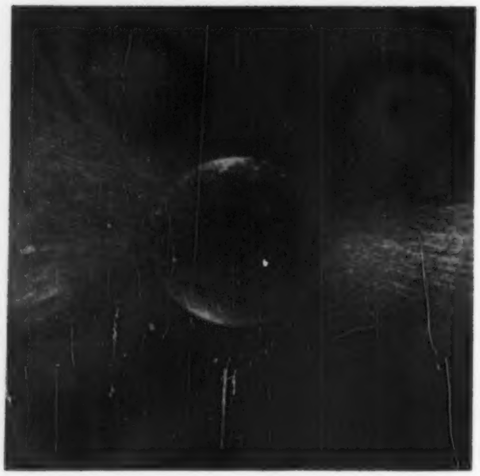
(a) *Multiple Impression Die.*—The die was machined from tool steel and five small impressions made in its surface with a 2 mm. diameter Brinell ball. After impressing, the die surface was polished flat and the impressions measured. Fig. 8 shows a photograph of the die surface and identifies the impressions and records their depths.

(b) *Single Impression Die.*—A single impression was made in this die by loading centrally with a 1½ in. diameter bearing ball. After loading, the die face was polished flat and the impression dimensions measured. Fig. 9 shows the details of this die.

Before each test the die and specimens were swabbed with a solvent to remove any gross contaminant present. The lubricants were then applied as detailed in Tables III and IV

TABLE IV.—TEST CONDITIONS WITH SINGLE IMPRESSION DIE

Specimen	Lubricant	Lubricant Application
1s	Nil	Nil
2s	1E	Thin smear to specimen surface
3s	1E	Applied to die face in sufficient quantity to ensure filling of impression and covering of die surface.
4s	5B	
5s	13B	
6s	19	



Impression diameter ≏ 0·250 in.
Impression depth = 0·0100 in.

Fig. 9.—Single impression die and details. × 4 approx.

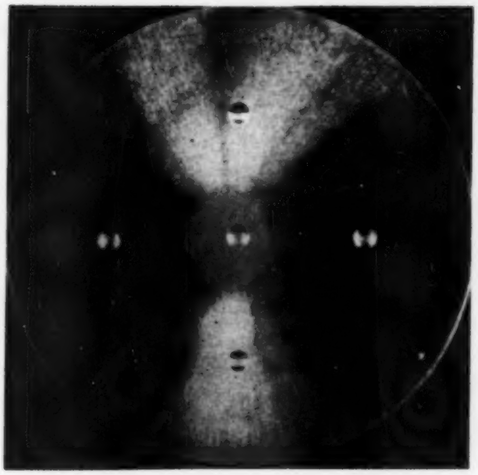


Fig. 10.—Specimen 1m.

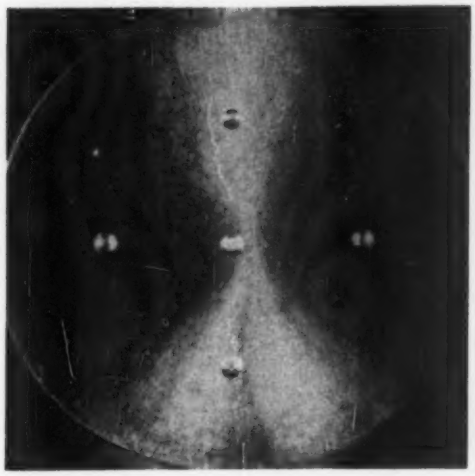


Fig. 11.—Specimen 2m.

Figs. 10-14.—Aluminium discs after deformation with various lubricants—see Table III. × 4 approx.

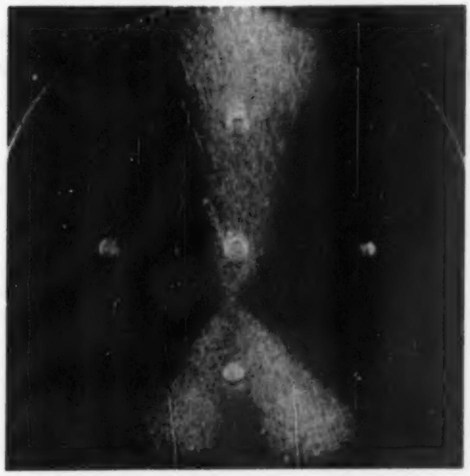


Fig. 12.—Specimen 3m.

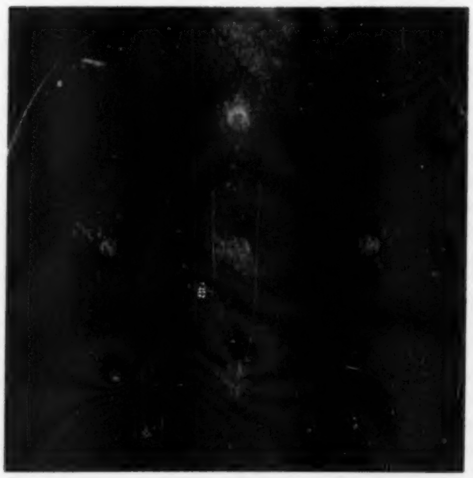


Fig. 13.—Specimen 4m.

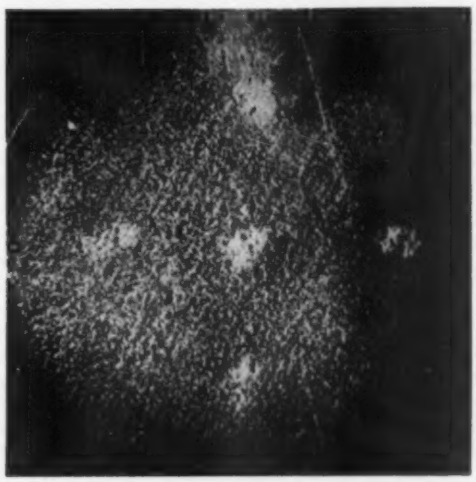


Fig. 14.—Specimen 7m.

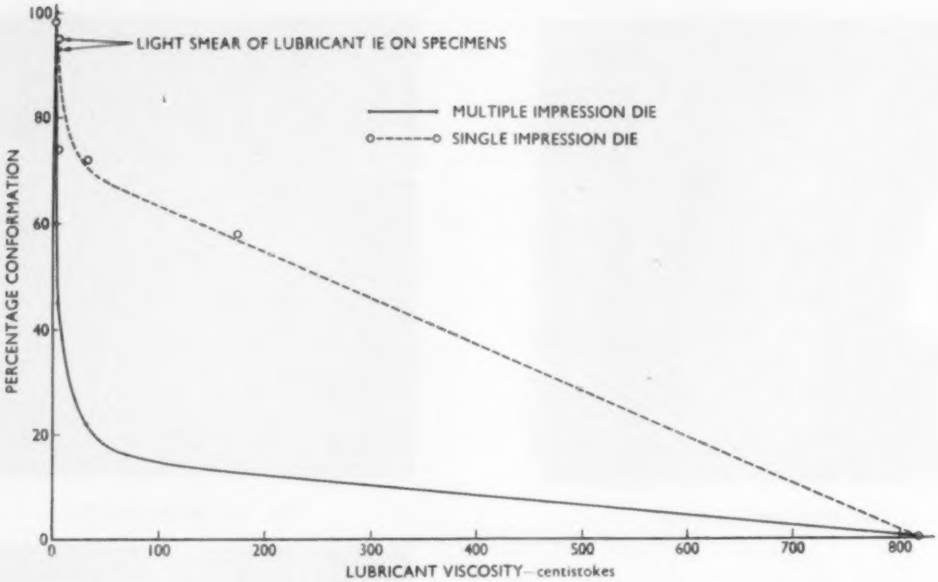


Fig. 15.—Percentage conformation v. lubricant viscosity. (Aluminium disc specimens: load 5 tons).

The specimens were rested centrally on the die face after the application of lubricant and a direct axial compressive load of 5 tons applied. This load was selected after having previously been established as sufficient to give complete specimen-to-die conformation in the absence of a lubricant.

During loading, dependent on the lubricant used, nipples were formed on the specimen surfaces corresponding to the impressions on the die surfaces. Although the nipples were not necessarily spherical in profile, a quantitative assessment of them was made by measuring their heights above the flat portion of the specimen surface plane. This enabled an arbitrary measurement of percentage conformation to be made. With the multiple impression die the maximum height of each of the five nipples on each specimen was compared with the depth of the corresponding die impression. The average of all five measurements was recorded as the percentage conformation of the particular specimen. For the single impression die, the percentage conformation was taken for each specimen as the ratio of the single specimen nipple height to the impression depth. The nipple heights and impression depths were measured both with a dial gauge reading to 0·0001 in. and by re-focusing a microscope under high magnification and measuring the stage travel. Readings consistent to within 0·0002 in. were obtained throughout.

The photographs in Figs. 10–14 of some of the specimen surfaces after loading, show with the multiple impression die that a large variation in surface conformation occurs with variation of lubricant viscosity. The nipples are well pronounced with no lubricant and with light lubricants, but become progressively less pronounced with the heavier lubricants. It is also apparent from Fig. 14 that with the heavier lubricants the additional effect of general surface roughening becomes serious. Here, even the portion of the specimen surface nominally in contact with the flat die areas, becomes matt and non-reflective. Fig. 15 plots the quantitative assessment of percentage conformation against lubricant viscosity and shows clearly how rapidly the conformation decreases with the increasing viscosity of the interposed fluid.

The results obtained using the single impression die were similar in character to those quoted above as shown in Figs. 15–19. Here, again, the presence of the lubricant has a marked effect on the conformation, and the general areas of surface disruption are clearly discernible on the nipples themselves and over the areas where expulsion of the lubricant from the die impression has occurred.

DISCUSSION

The experiments currently described were stimulated by previous observations[3,4] that in a simple compression test of a ductile aluminium disc between flat steel dies the surface appearance of the disc after compression was markedly influenced by the viscosity of the interposed lubricant. Even when polished and flat discs were used in conjunction with polished and flat dies—a surface condition apparently conducive to the complete expulsion of an interposed fluid before deformation started—it was found that a matt, non-reflective deformed surface was produced when using heavy lubricants. It was noticed, however, that a continuous thin, bright contact ring was formed around the contact periphery of the disc, which would probably prove an effective barrier to any trapped lubricant. The nature of the surface disruption, under inspection by microscope, was not that associated with cracking of the surface due to lateral expansion accompanying the axial compression. It was more a random disruption showing small discrete flattened areas, where contact with the die had occurred, interspersed with shallow troughs showing no die contact.

The following inferences may be drawn from these observations :—

(*a*) In the case of microscopically rough surfaces, the thin fluid films of lubricant filling the troughs between adjacent asperities became trapped between the surfaces on initial loading. Subsequent loading allowed

Fig. 16.—Specimen 1s.

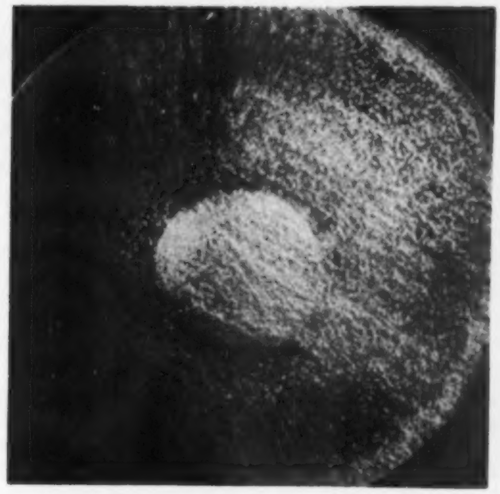


Fig. 17.—Specimen 3s.

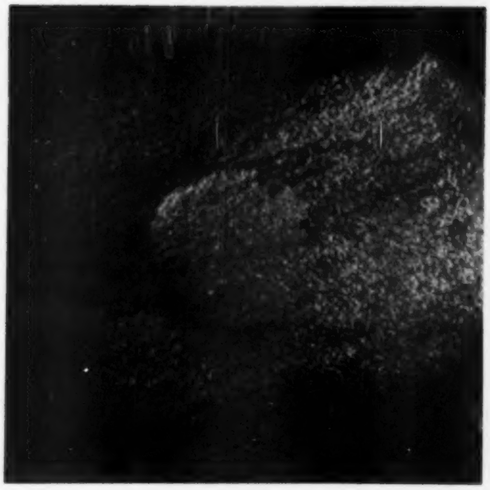


Fig. 18.—Specimen 5s.

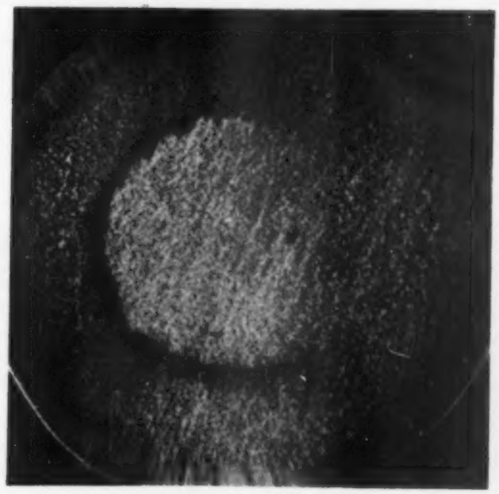


Fig. 19.—Specimen 6s.

Figs. 16-19.—Aluminium discs after deformation with various lubricants—see Table IV. × 4 approx.

some lateral escape particularly in the case of less viscous lubricants, until continuous peripheral contact between the die and disc was established.

(*b*) In the case of microscopically relatively smooth surfaces, a thin lubricant layer accepted all the initial load until peripheral contact occurred preventing its subsequent expulsion.

In either case it was assumed that continued deformation of the disc proceeded through die-to-disc contact at discrete points only, and largely via the fluid pressure built up in the entrapped film. Such a uniform fluid pressure acting over areas spanning several crystals could cause inhomogeneous deformation due to the varying orientations of the crystals, or cause slip within the crystals themselves, resulting in the production of a matt surface appearance.

It is accepted that the majority of liquids can withstand considerable pressure, usually with some increase of viscosity, without suffering much compression. The fact that mechanically entrapped fluids can help to cause large bulk plastic flow of metals has only infrequently been demonstrated. The current experiments show that this can easily be achieved under relatively arduous loading conditions. Both the experiments on the steel cones and those on the aluminium discs reveal that little, if any, expulsion of the heavier lubricants occurred after appreciable bulk plastic compressive strain (12% for steel and approximately 30% for aluminium). There seems little reason to doubt that the presence of fluid films such as have been deliberately maintained on a macroscopic scale can be achieved fortuitously on a microscopic scale as a result of the surface topography,

rheological properties of the fluid, and boundary sealing effect.

In practical metal forming processes this phenomenon of mechanical lubricant trapping is probably fairly common. Wistreich[5] has described experiments in wire drawing in which electrical resistance measurements between die and wire sometimes indicate only occasional contact. Christopherson[6] also has described another method of obtaining full film lubrication in wire drawing, although this was principally a hydrodynamic effect.

In cold strip rolling it is known that the use of a viscous base lubricant results in a matt surface appearance on the sheet, which may stem from the inclusion of semi-continuous lubricant films between the rolls and strip, which would cause inhomogeneous deformation of the strip surface.

Ford[7] has stated that the coefficients of friction as measured during deformation processes appear to be lower than those measured between similar metal pairs in more conventional sliding friction tests. In the presence of lubricants this fact may also be explained by the entrapped film. The area of the metallic junctions formed between die and metal is reduced in the presence of the film, and the subsequent frictional resistance results from the reduced metallic junction shearing component together with the component required to shear the lubricant film.

The tests currently reported have restricted the lubricants used to pure paraffinic base oils with no deliberate additives, since the lubricant viscosity alone was considered as the most important property likely to contribute to the establishment of trapped films. Such lubricants are of little use where boundary conditions prevail, as their boundary lubricating properties are poor, and would seldom be used alone in a metal forming process. The incorporation of boundary additives, however, whilst contributing to a reduction of the total metallic shearing resistance, would not affect the surface phenomena described unless they materially affected the rheological properties of the bulk fluid. Similar arguments apply also to lubricants of the " filler " type containing solid particles of inorganic material.

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Correspondence

Volumetric Determination of Magnesium in Aluminium Alloys

The Editor, Metallurgia.

Sir,

Dr. S. Bertoldi of the Fiat Laboratorio Ricerche e Controlli Auto-Avio, Turin, Italy, has commented on the following sentence in the above paper, published in Metallurgia[1] under the names of the undersigned :—

> " The details of the bromine separation have already been published in this journal by J. C. Sergeant from one of the Association's laboratories, whilst the zinc oxide technique has not previously been described in the literature."

He points out that the sentence is incorrect, as this technique has already been described by him[2], and at a still earlier date (unknown to Dr. Bertoldi at the time of his publication) in a paper by G. S. Smith.[3]

Dr. Bertoldi's interpretation of the sentence referred to is, we consider, a misunderstanding insofar as we did not intend to claim the authorship of the zinc oxide technique as such, any more than that for the well-known bromine-sodium-acetate separation can be claimed by Sergeant. What was meant was rather that the application of a well-known technique to the EDTA finish had not previously been published. In some British laboraratories, in particular in the laboratory of one of us (R.C.J.), the precipitation of iron, aluminium and manganese in the way described, which ultimately derives from the well-known method of Volhard and Wolff,[4] had been in regular use for many years prior to the introduction of EDTA, and it was introduced into the latter laboratory following a paper by R. Bauer and J. Eisen[5] in which, by the way, no reference is made to the work of G. S. Smith, nor to that of Dr. Bertoldi and his associates.

We now take pleasure in acknowledging, with apologies, the priorities mentioned above.

Yours faithfully

R. C. Jackson, W. Stross,

L. H. Wadsworth.

1 *Metallurgia*, 1958, **58** (350), 305.
2 Prever, V., Bertoldi, S., and Bargagliotti, A., *Alluminio*, 1938, **7**, 247.
3 *Analyst*, 1935, **60**, 812.
4 *Ann. Chem.*, 1879, **198**, 318.
Dr. Bertoldi in his paper had mentioned these authors.
5 *Aluminium*, 1941, **23**, 290.

We regret the delay, due to an oversight, in the publication of this letter, and trust that no inconvenience has resulted therefrom.—*Editor*.

Alcan Smelter Expansion

Aluminum Co. of Canada, Ltd., recently announced that $1,630,000 will be invested at their Kitimat, British Columbia, smelter for furnaces and other equipment to provide aluminium fabricators at home and abroad with increased tonnages of extrusion ingot. According to Alcan, the equipment to be installed reflects the growing market for aluminium in forms which an extruder can immediately fabricate. The installation of this new equipment will enable Alcan to serve this market better. The new equipment at Kitimat will be in operation by the end of 1960 and will enable the plant to produce some 36,000 tons of extrusion ingot annually out of its present rated capacity of 186,000 tons of primary aluminium.

T.I. Acquisition

Tube Investments, Ltd., has acquired the whole share capital of Fords (Finsbury), Ltd., of Bedford. The advanced precision engineering products of this company, particularly in the field of high-speed equipment for capping bottles and forming other aluminium closures, provide close ties between the engineering and aluminium interests of T.I. and Fords (Finsbury), Ltd. Mr. R. E. Ford will continue as chairman of the board of the company and its other directors will continue in their present appointments.

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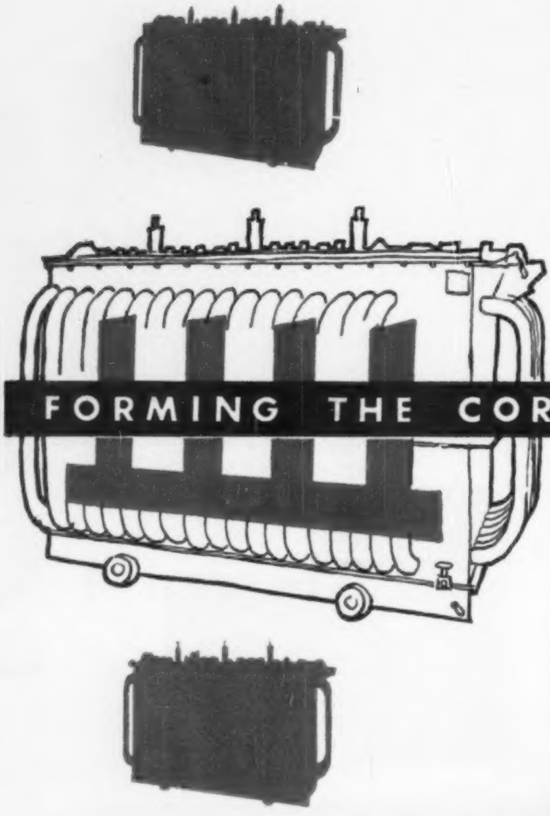
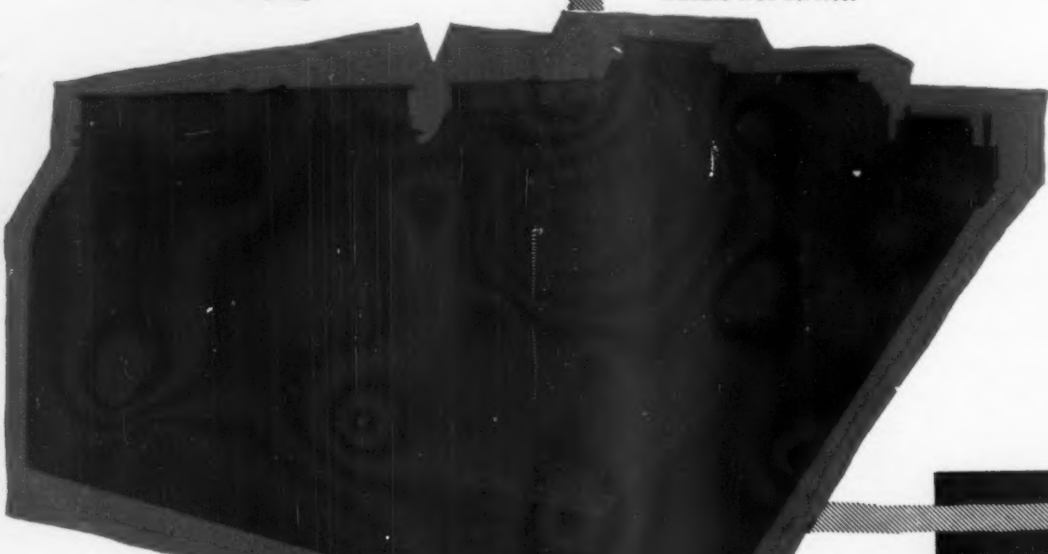


FORMING THE CORE

These 180 kW Birlec bell furnaces at the works of Joseph Sankey & Sons Manor Works Ltd., Wolverhampton, have been installed for the high temperature annealing of hot-rolled silicon steel sheet for transformer cores. Two more Birlec installations, totalling a further ten bell furnaces, form a substantial part of Sankey's annealing capacity. Some 450 tons of transformer sheet, with a finished value exceeding £36,000, are treated each week in Birlec Furnaces.

** The cost of a furnace is not necessarily its purchase price. Even a brief interruption in production may cause severe losses in output and serious inconvenience. The purchaser of a Birlec furnace can be confident that the equipment will not only meet his specification, but will give uninterrupted trouble-free service.*



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NEWS AND ANNOUNCEMENTS

Symposium on Wide Strip

WITH new mills being built in Scotland and South Wales, wide steel strip production is a subject that is being studied very closely by the U.K. iron and steel industry at the present time. It is appropriate and timely, therefore, that The Iron and Steel Institute should devote the technical sessions at its Annual General Meeting this year to a symposium on wide strip production.

The scope of the symposium is wide, covering all aspects of strip production from steelmaking to manufactured goods. The opening session will review the future requirements of the tinplate and vehicle industries, the major users of wide strip. This will be followed by a session devoted to steelmaking practice, with papers from the Steel Company of Wales, National Steel Corporation (U.S.A.), Cornigliano (Italy), and Hoogovens (Holland); the subjects under discussion include converter processes, oxygen in the open-hearth, and quality control. Strip rolling is dealt with in the third session, with papers from Davy-United and United States Steel Corporation. The fourth session is a mixed bag, with papers on the metallurgical principles of tinplate manufacture (S.C.O.W.), rolling of very thin tinplate (United Engineering and Foundry Corporation, U.S.A.), high-speed rolling (John Summers), and annealing (Lysaghts, Australia, and S.C.O.W.). The final session deals with coatings—tin (S.C.O.W.), galvanizing (John Summers), and plastics (B.I.S.R.A.).

The symposium starts at the Royal Commonwealth Society, Northumberland Avenue, London W.C.1, on the afternoon of Tuesday, 3rd May, 1960. The subsequent sessions, on 4th and 5th May, will be held in the Hoare Memorial Hall, Church House, Great Smith Street, London S.W.1.

Heat Treatment Conference

A CONFERENCE on heat treatment practice, organised by the British Iron and Steel Research Association, will be held at Harrogate on 5th and 6th July next. The main aspect of the subject to be considered will be "carburising," the various papers ranging over the theory and practice of carburising and the properties of carburised steels. Full organisational details and registration forms will be available from 6th May; applications should be made to the Technical Secretary, Metallurgy (General) Division, The British Iron and Steel Research Association, 11 Park Lane, London, W.1.

Open Days at N.E.L.

THE National Engineering Laboratory is to hold Open Days on Wednesday and Thursday, 15th and 16th June, 1960, when the laboratory will be open for inspection. Special displays have been arranged including many items developed since the last Open Days in 1958. Facilities for sponsored investigations for industry will be on show, as well as results of work carried out as part of N.E.L.'s general programme of basic and applied research. These include the automatic measurement of errors in machine tools, the development of hydrostatic transmissions, research on the cold extrusion of steel, studies of the performance of high-speed bearings, investigations of the growth of fatigue cracks, and the preparation of new international steam tables.

Representatives of any organisation with engineering interests will be welcome. Applications for invitations, stating which day is preferred, should be sent to: The Director, National Engineering Laboratory, East Kilbride, Glasgow.

U.S. Visit

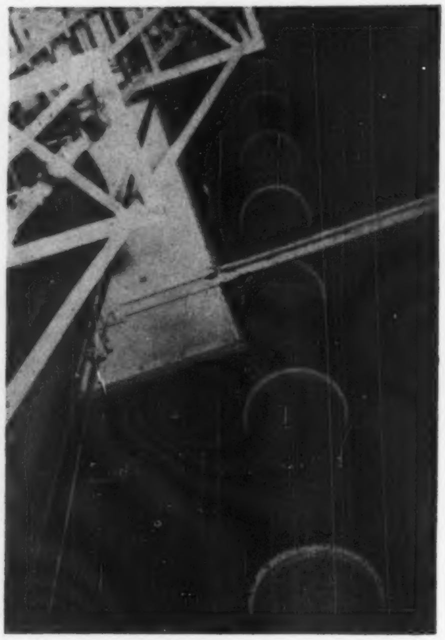
DR. W. E. HOARE, assistant director of the Tin Research Institute and a world authority on tinplate, is at present on a month's tour of the U.S.A. and Canada, where he is visiting a number of important tinplate and canmaking centres, and particularly their associated research and development establishments. Notable topics for discussions will include the development of quality appraisal techniques suited to the high grade tinplate of modern commerce; the increasing use of light gauge tinplate; and the development of shipment in coils rather than in sheets.

The Tin Research Institute has recently interested itself in the use of tin as a structure controlling addition to engineering cast iron. A considerable amount of investigational work has been carried out on the Institute's behalf at the Battelle Memorial Institute at Columbus, Ohio, and at the Institute's Headquarters and laboratories in London. Dr. Hoare's itinerary includes some iron foundries which are already using the technique.

Nickel Alloys Exhibition

FOR the second time Henry Wiggin & Co., Ltd., has held a private exhibition in London to help engineers and designers to make the best use of the company's specialised high-nickel alloys. In the 125 years of its existence, the character of the business has changed with the times, and today the emphasis is very much upon high nickel-containing alloys of complex compositions and produced to even more complex specifications. Experience has shown that while many of their customers are fully conversant with the use of Wiggin alloys in the products which they themselves manufacture, they are not always aware that in the components of equipment and plant used in their own processes other nickel and nickel alloy products also play a prominent part, and the exhibits on display were selected with these points in mind.

Included in the items on show were examples of Wiggin alloys in the aircraft industry, including a Nimonic 80A catalyst pack-retaining grid for a Super-Sprite rocket motor by the de Havilland Engine Co., Ltd., and the disc and blades of a gas turbine rotor employing Nimonic creep-resisting alloys by Bristol Siddeley Engines, Ltd. The marine field included an example of a variable pitch propeller with blade roots of K Monel by Slack and Parr (Marine), Ltd., and under the heading "Chemical Plant" was included a hot-pressed dished head in Corronel 210 by G. A. Harvey and Co. (London), Ltd. In the electrical field was shown a Saferod heater with an Inconel sheath enclosing the Brightray C element by Heatrae, Ltd., and in electronics, as an example, was included the printed circuit of an Avonmeter made in Ferry resistance alloy, by Technograph Printed Circuits, Ltd.



A section of the Stelvetite ducting installed inside an existing 270 ft. reinforced concrete stack at Avonmouth.

The Nimonic series of alloys have played a big part in the outstanding success of British aero engines, and it is understood that yet another member of the series with enhanced high temperature properties is to be announced soon. Furthermore, these alloys are now finding increasing use in industrial applications on account of their remarkable strength at high temperatures, and the new equipment installed at Wiggins at Hereford will enable them to offer a wide range of forms required in modern engineering practice.

Stelvetite in New Beryllium Plant

STELVETITE, the plastic-coated sheet steel, forms the interior of the P.V.C./steel/reinforced epoxy resin laminate used for a ventilating shaft fabricated by Ductwork, Ltd., and resin coated by Parglas, Ltd., and installed inside an existing 270 ft. tall reinforced concrete stack at the works of the National Smelting Co., Ltd., Avonmouth.

Stelvetite was suggested by Parglas Ltd., as the most economical means of providing the basis of a rigid structure which, with its internal lining of P.V.C., would be resistant to the traces of acidity present in the filtered ventilation gases, the external applied coating of epoxy resin being resistant to the traces of ammonia in the surrounding atmosphere. Produced by John Summers & Sons, Ltd., of Shotton, Chester, Stelvetite can be bent, formed, deep drawn and welded without damaging the coating.

The structure forms part of the new beryllium plant which has been in production since November. This is the only commercial operating plant extracting beryllium in the U.K. It provides this metal for sheathing uranium fuel elements in nuclear reactors.

Reverbale Junior aluminium melting furnace

Reverbale Furnaces in Canada

THE ENGLISH ELECTRIC CO., LTD., of St. Catherine's, Ontario, Canada, have recently taken delivery of two Reverbale Junior furnaces, supplied through Messrs. Drew Brown, Ltd., Canadian agents for Sklenar Furnaces, Ltd., of Cardiff. It is interesting to note that these furnaces were introduced to English Electric by one of their executive engineers who got to know the Sklenar Reverbale in the English Electric factory at Bradford, Yorks.

The Reverbale Junior, a highly compact and efficient furnace for melting aluminium alloys, is made in two sizes: Type I, with a capacity of 150 lb. of aluminium, and Type II, holding 250 lb. Rate of melting for both types is 300 lb./hr., which is made possible by the patented design of the refractories which ensures maximum thermal efficiency. This, coupled with the use of waste gas for preheating, ensures rapid melts with low fuel consumption. Indeed, the claim is made that melting and maintenance costs are reduced by as much as two-thirds as compared with crucible practice. The characteristics of the Reverbale and Reverbale Junior furnaces make them popular units with light metal die-casters; in the electrical industry they are frequently used for high conductivity castings, alloy castings for motor housings, and for domestic appliances.

The Determination of Gases in Metals

IT has long been recognised that the minute traces of the gases oxygen, hydrogen, and nitrogen in a metal can significantly affect its mechanical properties and its service performance. In order to control these gaseous impurities, they must be measured accurately, and many new determination techniques have been evolved in recent years. A forthcoming symposium on "The Determination of Gases in Metals," organised by the Society for Analytical Chemistry in conjunction with The Iron and Steel Institute and The Institute of Metals, will review the present position, and will survey the latest techniques available. In addition to authoritative

review papers covering each of the three gases, there will be papers on vacuum-fusion, carrier-gas, activation analysis, emission spectrometric, and internal friction methods of determination from experts in these fields. The symposium will be held at Dennison House, 296 Vauxhall Bridge Road, London, S.W.1, on Tuesday and Wednesday, 3rd and 4th May, 1960.

Electric-Arc Steelmaking

It was recently announced that the open-hearth melting shop at Steel, Peech and Tozer, Rotherham (a branch of The United Steel Cos., Ltd.) was to be converted to an electric-arc melting shop, the largest in the U.K. A paper on the new installation by Mr. A. Jackson, technical adviser on steelmaking to United Steel, and Mr. R. S. Howes, melting shop manager, Steel Peech and Tozer, is being presented at the Annual General Meeting of The Iron and Steel Institute on the morning of Thursday, 5th May, 1960. The paper gives the reasons for taking this bold decision, and surveys the equipment of the new shop. The meeting will be held at the Convocation Hall, Church House, Great Smith Street, London, S.W.1. starting at 10.30 a.m. Admission is free.

Rectifiers for Tinplate Expansion

An order valued at more than £200,000 has been received by A.E.I. Heavy Plant Division from the Tinplate Division of the Steel Company of Wales for germanium rectifiers for a third tinning line at the company's Trostre Works at Llanelly. The equipment, which will consist of five large groups of rectifiers, will be used for the cleaning, pickling, tinning, and chemical treatment of strip in a range of widths from 27 in. to 38 in., and thicknesses from 0·006 in. to 0·015 in. The total capacity of the installation will be approximately 4,000 kW. and the aggregate current more than 200,000 A. at voltages up to 24.

The two largest groups of rectifiers will be used for the tinning operation. Saturable reactors provide independent control of the output of the rectifiers to each side of the strip, and in this way the application of the tin can be controlled. For canning purposes, tinplate needs a heavier coating of tin on one side (the inside of the can) than the other, so during the tinning process the output of the rectifiers is adjusted to take this requirement into account. Air-cooling is used, heat exchangers supplying cooled air to the germanium cells in an enclosed forced-ventilation system.

Consultants for Indian Foundry

F. H. Lloyd & Co., Ltd., steelfounders and engineers, have secured a contract—which had been put out to world tender—to act as consultants to the Indian Railway Board, for the design, erection of buildings, installation of plant and equipment, and training of personnel for a steel foundry to be built at Chittaranjan in West Bengal. Its function will be to produce the steel castings required for locomotive construction to be carried out at Chittaranjan. Initially the annual output will be 7,000 tons for the planned construction of 200 locomotives.

The new foundry will be situated adjacent to the main assembly bays of the locomotive works, and will cover an area of 164,000 sq. ft. The main buildings will accommodate melting, coremaking, light and heavy foundry and dressing shops, heat treatment, inspection and despatch, and auxiliary buildings will provide for X-ray and other non-destructive testing facilities, sand and metallurgical field laboratories, lockers and washrooms, electricity supply, sub-station, etc.

The light and medium foundry will be equipped with moulding machines—slinger rollover units—and the heavy foundry, dealing mostly with floor and pit moulding, will make use of travelling sand slingers. Molten steel will be provided by two electric arc melting furnaces, and the main handling equipment will consist of electric overhead cranes.

F. H. Lloyd & Co., Ltd., will be responsible for the layout of the buildings and plant, layout of all services, the preparation of specifications for plant and equipment, erection and installation.

The manning of the foundry is a feature that will require careful planning. Initially, after design, selection of plant, and machinery has been decided, a site engineer will go to India and be responsible for the erection of the buildings and the installation of plant.

Kent Production Concentration

With the transfer on 2nd April, of all the interests, assets and production capacity of their steering gear division to the ownership and management of Cam Gears, Ltd., George Kent, Ltd., will now devote their manufacturing and management capacity exclusively to their main interests of industrial instrumentation and process control, and mechanical meters. For more than thirty years Kent have been manufacturing steering gears, in their position as the sole bulk suppliers to Cam Gears, Ltd., through which company alone they have had any contact with the motor trade. The growing main instrumentation and meter business of the Kent company will continue to be transacted from the factories in Luton and the Kent subsidiaries and representations about the world. The growing complexity and demand of the process-control field has weighed considerably as an inducement towards this rationalization of the business as a whole and it is confidently expected that the present pace of planned technical and sales development will now be increased.

U.S.S.R. Patents Report

A patents delegation visited the U.S.S.R. in November/December last year, and on their return an interim report was published in the *Board of Trade Journal* on 25th December (p.1045). The final report of the delegation, which also contains translations of the current Soviet Laws and Instructions to applicants for patents and trade marks has now been published. It can be obtained from H.M. Stationery Office at the usual addresses or from the Sale Branch, Patent Office, 25, Southampton Buildings, Chancery Lane, London, W.C.2. price 2s. 6d. (by post 2s. 10d.).

Pipes and Pipelines Exhibition

When the sponsors of the first International Pipes and Pipelines Exhibition, which is due to be held at Earls Court, London, from May 30th to June 2nd 1960, first publicised their intention to hold this exhibition they had reserved a hall giving 14,000 sq. ft. of stand space. However, in view of the overwhelming support that immediately resulted, the area reserved had to be subsequently increased to 23,000 sq. ft. of stand space, and as

this area was also rapidly filled by the reservations of both British and overseas exhibitors, the total area of stand space has had to be increased once more to 28,000 sq. ft.

Besides a large number of exhibitors who will be showing different types of pipe materials, many stands will show important ancillary equipment such as insulation, protection, testing and welding equipment, as well as fabricated pipework, pumps, valves and fittings. Tickets can be obtained by application to the Organisers : Scientific Surveys Ltd., 97, Old Brompton Road, London, S.W.7., England.

I.S.I. Medals and Prizes

THE Council of The Iron and Steel Institutes has announced the award of the following medals and prizes :

Bessemer Gold Medal for 1960 : To Professor DR. HERMANN SCHENCK, Director of the Institut für Eisenhüttenwesen, Rheinische-Westfälische Technische Hochschule, Aachen, Germany, and President of the Verein Deutscher Eisenhüttenleute.

Sir Robert Hadfield Medal for 1960 : To DR. J. C. HUDSON, Head of the Corrosion Section, Chemistry Department, British Iron and Steel Research Association.

Andrew Carnegie Silver Medal for 1959 : To DR. P. R. V. EVANS, formerly Research Department, Metropolitan-Vickers Electrical Co., Ltd., Manchester, for a paper on "The effect of rolling unstable austenitic 0·76% carbon steel at 220-300° C." (*Journal*, 1959, January, p. 34) ; his co-author, Professor Hugh O'Neill, was not eligible for an award.

Williams Prize for 1959 : To MR. I. M. D. HALLIDAY, Research and Development Department, The United Steel Companies, Ltd., Rotherham, for a paper on "Continuous casting at Barrow" (*Journal* 1959, February, p. 121).

These awards will be made at the Institute's Annual General Meeting at the Royal Commonwealth Society, Northumberland Avenue, London, W.C.1. on the morning of Tuesday, 3rd May, 1960.

Spring Welding Meeting

NEW welding processes are constantly being developed to meet the ever more exacting demands of the engineering industries. The Spring Meeting of the Institute of Welding, to be held this year at Droitwich from 9th-11th May, is to be devoted primarily to some of the more recently developed processes, such as fine wire welding, electro-slag welding and high frequency welding. In addition, one whole session is to be given to a group of papers on the varied uses of metal spraying.

Telcon Metals Ltd.

BRITISH INSULATED CALLENDER'S CABLES, LTD., announce the formation of a new company, Telcon Metals Ltd. This company, centred at Crawley, has assumed responsibility for all the activities of the Metals Division of The Telegraph Construction and Maintenance Co., Ltd. These cover the manufacture of the well-known Telcon magnetic materials and other special alloys used in the electrical and engineering industries and also the advanced metallurgical operations associated therewith.

The board of directors of the new company will consist of : MR. W. C. HANDLEY (chairman), MR. W. F. RANDALL (deputy chairman and managing director), DR. G. A. V. SOWTER (commercial director), DR. H. H. SCHOFIELD (technical director), and MR. D. NORMAN-THOMAS, T.D. MR. L. D. DODD will be secretary.

In addition to operating the factory at Crawley, Telcon Metals, Ltd., will control the following subsidiary companies : Magnetic and Electrical Alloys, Ltd. (Burbank, Hamilton, Lanarkshire) ; Telcon-Magnetic Cores, Ltd. (Chapelhall, Lanarkshire) ; Temco, Ltd. (Lydbrook, Glos.) ; and Toolpro, Ltd. (Ilford, Essex).

Lord Hailsham Visits B.I.S.R.A.

LORD HAILSHAM, the Lord Privy Seal, visited the Hoyle Street laboratories of the British Iron and Steel Research Association on Wednesday, March 30th, in the course of his tour of the Sheffield area. In view of his special responsibility for scientific matters, the Department of Scientific and Industrial Research arranged the B.I.S.R.A. visit, in conjunction with the Association. Lord Hailsham, who was accompanied by Sir Harry Melville, secretary of the D.S.I.R. was received by Dr. Charles Sykes and Sir Charles Goodeve, chairman and director of B.I.S.R.A., respectively, and Dr. J. Pearson, assistant director and head of the Hoyle Street Laboratories. The visitors saw a number of B.I.S.R.A. projects, which included the work on vacuum melting, the "APIC" device (automatic power input control) for arc furnace use ; and the recently announced automatic forging project. The opportunity was also taken of visiting the laboratories of the Coil Spring Federation Research Organisation, the Cutlery Research Council and the File Research Council, which occupy premises on the same site and which have close ties with B.I.S.R.A.

I.S.I. to Visit Italy

THIS year's Special Meeting of The Iron and Steel Institute, from 29th May to 11th June, 1960 will be held in Italy for the first time since 1923. The meeting will open with a two-day joint meeting with the Associazione Italiana di Metallurgia, at which Mr. W. F. Cartwright, who takes office as President of the Institute at the A.G.M. on 3rd May, is to be awarded the Giolitti Medal of the A.I.M. one of the highest honours that body can award. There will also be joint technical sessions dealing with direct reduction, continuous casting, and vacuum casting.

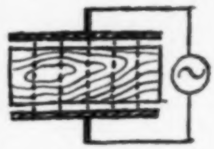
Following the joint meeting, members of the Institute will visit many of the leading iron and steelmaking plants in Italy, including Breda, Cornigliano, Dalmine, Falck, FIAT, Ilva-Bagnoli, Innocenti, Nazionale Cogne, S.I.A.C., San Eustacchio, and Terni.

A.L.A.R.S. Annual Meeting

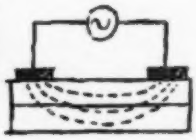
AT the second Annual General Meeting of The Association of Light Alloy Refiners and Smelters, Ltd., held on 5th April, 1960, MR. W. W. KEE (Enfield Rolling Mills (Aluminium), Ltd.), was elected chairman and MR. R. HAHN (B.K.L. Alloys Ltd.), was elected vice-chairman, to hold office until the end of the next Annual General Meeting. The council of management for the same period consists of the chairman, the vice-chairman, and the following members : MR. F. FARENDEN (The Eyre Smelting Co., Ltd.), DR. J. JAKOBI (International Alloys, Ltd.), MR. L. A. JARVIS (Wigley Aluminium, Ltd.), and MR. P. WARD (John Dale, Ltd.).

Dielectric Heating - 1

When an electrically non-conducting material is placed between two metal plates, called electrodes, connected to an A.C. supply, the alternating electrostatic field between the electrodes considerably speeds up the molecular movements in the material (termed a 'dielectric') as a result of which the temperature of the material under treatment rises. A similar effect is produced where the two electrodes are positioned on the same side of the dielectric; in this case the electrostatic field between them is generally known as a 'stray' or 'fringe' field.



For industrial application, the applied voltage of the order of 15,000 volts supplied by an electronic generator alternates at frequencies of some millions of cycles per second.



The amount of heat generated in the dielectric is determined by the frequency, the square of the applied voltage, the dimensions of the object and a physical property of the material termed "loss factor" and is represented by the equation:—

$$\text{Power} = 1.41\, E^2 f.\, F\, \frac{A}{t} \times 10^{-15} \text{ kilowatts.}$$

Where E = applied voltage, f = frequency, F = loss factor.

A = area of the dielectric in square inches.

t = thickness of the dielectric in inches.

F, the loss factor, is itself equal to the expression K Cos Ø in which:
K = the dielectric constant, a measure of the property of the material to retain energy arising from disturbance of its molecular structure.

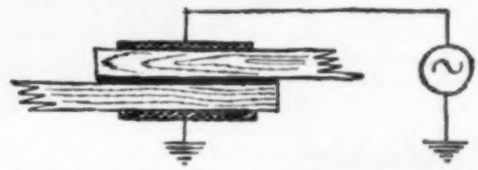
Cos Ø = the dielectric power factor of the load, that is the ratio of the power (in watts) to the product (in volt-amperes) of the voltage and current. This is a characteristic property of the material.

Therefore, "loss factor" is a property of the material and a measure of the ease with which it can be heated by this method. Like other physical properties, it varies considerably for different substances. The equation shows that the heat generated in a dielectric is proportional to its loss factor, but the rate of rise of temperature will also depend upon its specific heat and density. The following table gives approximate values of the dielectric constant, power factor and loss factor of a few typical dielectric materials for frequencies around a million cycles a second.

MATERIAL	DIELECTRIC CONSTANT	POWER FACTOR	LOSS FACTOR
Natural Rubber	2.9	0.02	0.058
Oak, dry	3.3	0.04	0.132
P.V.C.	5.3	0.06	0.318
Urea formaldehyde	7.0	0.03	0.21
'Bakelite' resin	6.0	0.03	0.18
Nylon	3.7	0.05	0.185
Water, pure	80.0	0.03	2.40
Water, tap	80.0	0.5/5.0	40/400

The high loss factor of water means that materials which are difficult to heat when completely dry will often heat efficiently when moisture is present. The voltage must be increased towards the end of the process in some cases to remove the final moisture traces, the reduction in loss factor as the material dries out providing a safeguard against overheating.

Dielectric heating of a homogeneous material is a straightforward application, heat being generated uniformly throughout. If the workpiece is made up of a number of materials, each material will heat up uniformly but each at a rate depending upon its loss factor, thermal properties and density. The degree of temperature uniformity throughout the workpiece will then depend upon the extent to which thermal conductivity can equalise different rates of heating.



Such different rates of heating can be turned to good account in certain applications. For example, in wood glue setting, the glue lines heat up much more rapidly than the wood pieces being joined and the glue sets before the wood heats up substantially, wood having a lower loss factor than glue. Dielectric heating does not depend upon any external heat source to transfer heat by conduction, convection or radiation to the surface of the charge and from thence to the interior by conduction.

Instead, heat is generated within every particle of a body placed in the dielectric field and, depending upon the uniformity of such a body, an even and extremely fast temperature rise can be achieved.

RECENT DEVELOPMENTS
MATERIALS : PROCESSES : EQUIPMENT

Bellows Type Flowmeter

A NEW bellows type flowmeter featuring automatic temperature and static pressure stabilising—introduced by Honeywell Controls, Ltd.—is claimed to be the most stable in operation of its kind yet developed. It will operate efficiently with ambient temperatures between $-40°$ F. and $+250°$ F., and it incorporates a rapid pulsation damping device. The instrument, which is suitable also for liquid level measurement, has an accuracy of $\pm 0\cdot5\%$ full scale differential pressure. Positive overload protection is provided.

Fifteen different ranges from 0–20 in. water to 0–400 in. water are available, and only the simplest component change is necessary for range changing. A large bellows system and torque tube assembly give high torque and power output for the operation of additional elements. The flowmeter can be used in conjunction with integrally mounted or remote reading indicators, recorders or controllers, and automatic flow totalising on a six digit counter can also be incorporated.

The bellows and all parts of the bellows assembly exposed to the process fluid are of stainless steel, making the flowmeter suitable for use with almost any process fluid. Ease of installation is assured by pressure connections for vertical and horizontal piping, which can be positioned at either the top or the bottom with no change of parts.

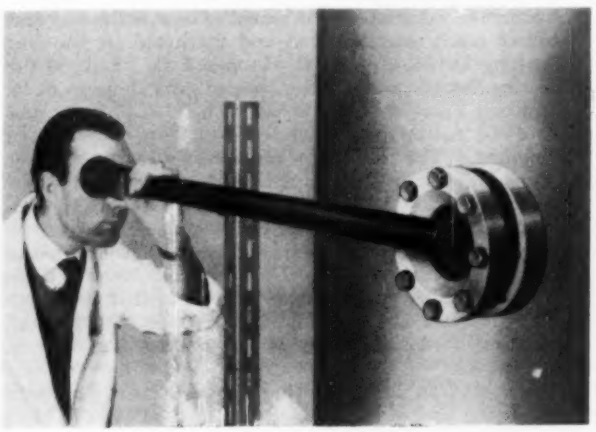
Honeywell Controls, Ltd., Ruislip Road East, Greenford, Middlesex.

Safety Periscope

A SAFETY periscope has been added to the extensive range of remote and indirect viewing equipment made by P. W. Allen & Co., a company specialising in lighting and optical equipment for visual inspection and observation in industry and research. The new instrument, known as the Allen Type A.201, is for the use of observers when closely viewing equipment and processes through sight glasses in pressure vessels, furnaces, chemical plants, etc., who are in danger of serious injury in the event of window breakage. Using the periscope the observer can stand well clear of the window in a safe zone and yet have his " eye " right up to the sight glass.

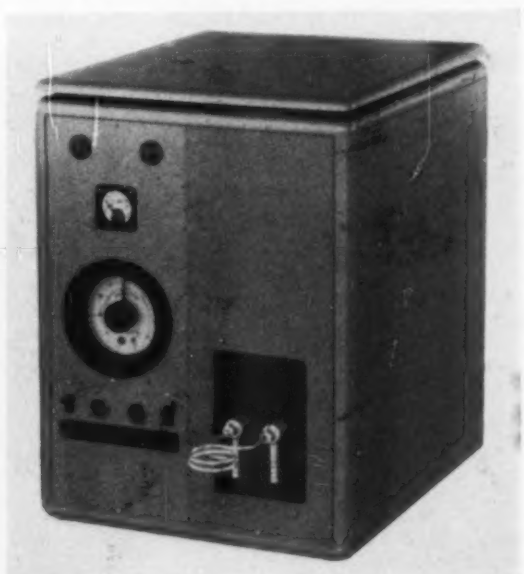
The Allen A.201 consists of a periscopic optical system of unit power with a field of view of 35°, fully corrected and free from distortion. The optics are contained in a metal tube 30 in. long and 1½ in. diameter fitted at one end with a rubber eye shield and focusing sleeve and at the other a 90° prism. Objects between 3 in. and infinity from this prism can be focused, with considerable depth of focus at any setting. A 90° eyepiece is available for use when the periscope has to be held horizontal at a level lower than the eye. Low voltage high intensity spot lights are also available to fit on the end of the periscope for such uses as when viewing equipment inside pressure vessels which are without internal lighting.

P. W. Allen & Co., 253, Liverpool Road, London, N.1.



Induction Heater

THE second induction heater in the new Pye range is the R.F.2, which is a medium impedance unit and gives a continuous output of 1·5 kW. at 2 Mc./s. It has been designed for bench operation, and is similar in appearance to the R.F.2, having an aluminium cubicle finished in green-grey hammertone enamel, with safety switches on the removable sides : the overall dimensions of the unit are 24½ in. high × 17½ in. wide, × 21¼ in. deep. The equipment is self contained with its own automatic resetting process timer, overload relay to protect the valves, and water pressure switch to protect the equipment. Provision is made for remote control, and a pulse



is available at the end of the heating cycle to initiate a quench or any other operation required. The equipment is designed to be operated from a single phase supply, 180–250 V., 50 c./s., with a full load consumption of 3·1 kW.

Pye, Ltd., R.F. Heating Division, 28 James Street, Cambridge.

Aluminium Foil Wound Transformer

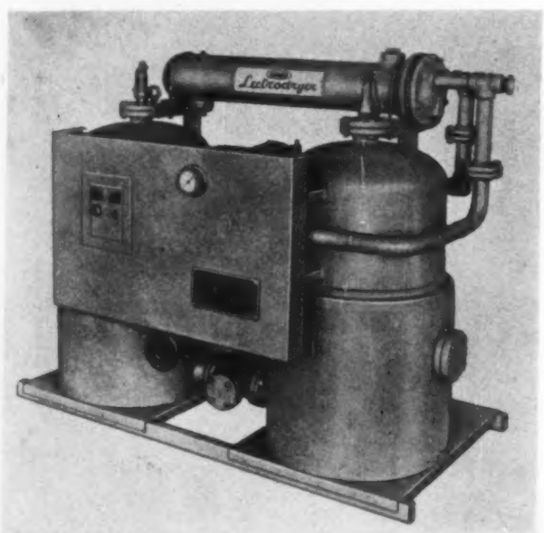
The Balarc 175 welding transformer is believed to be the first design ever wound in aluminium foil, and as a result it realises a high power-to-weight ratio. Electrolytic treatment of the foil allows temperature rises in the region of 500° C., which is far beyond that possible with copper coils. New designs of formers and insulation have made this Balarc 175 transformer a robust and most durable air-cooled welding unit weighing only 67 lb. for easy transportation.

This new unit is rated for 175 A. continuous operation, and meets the specifications of many countries (including the United Kingdom, U.S.A. and Germany). It is ideal for continuous operation in high ambient temperatures and adverse climatic conditions, each unit being chemically treated to prevent the ingress of moisture or foreign bodies. A two-pole, six-way roller switch controls amperage at both 80 and 50 V. open circuit at 23 V. welding, running from 190 to 250 V. A.C. single-phase 50 c./s., and draws a maximum of 40 A. from the mains.

Ashton-Young Engineering Development, Co., Ltd., 76 Cambridge Road, Kingston-on-Thames, Surrey.

Electric Drying Equipment

G.W.B. Furnaces, Ltd., are now marketing a complete range of electric drying equipment under the name "G.W.B.-Lectrodryer". The basic element of the G.W.B.-Lectrodryer is an adsorber unit, containing a granular material, such as alumina, which adsorbs moisture from the air, gas or organic liquid passing through it. Subsequent heating by embedded heating elements or by a stream of pre-heated air restores the adsorbent to its original efficiency as a drying agent.



Single adsorber driers are suitable for intermittent operation, where drying is required only during the working day and reactivation can be effected at night. For continuous operation, units with twin adsorbers are used, one drying whilst the other is reactivated. These units can be equipped for manual switching of adsorbers, or for completely automatic operation. A feature of the adsorbing agent is that it is almost permanent, since despite continuous usage, the activated alumina does not "wear out".

A wide range of driers is available, from the 68 lb. model, handling up to 20 cu. ft./min. of air, to large custom built models for special applications. Auxiliary equipment, including devices for measuring moisture content and filters to prevent contamination by oil vapours, are also available. Applications for which the G.W.B.-Lectrodryer are specially suited include air conditioning, drying compressed air, drying helium for instrument filling, and hydrogen for sintering furnaces, and in wave guide dehumidification.

G.W.B. Furnaces Ltd., Dibdale Works, Dudley, Worcs.

New Design of Motor

An addition to their range of industrial motors is announced by the Motor and Control Gear Division of Associated Electrical Industries, Ltd. The new motor is designated Type KN-D, and is of the squirrel-cage induction class, totally enclosed fan-cooled, with ribbed frame and endshields. Class "E" insulation is used, permitting a maximum temperature rise of 65° C. The machine complies electrically with B.S. 2613 : 1957 and its dimensions are such that it is completely interchangeable, rating for rating, with the ventilated (AEI Type KN-C) British Standard Dimension motor built in accordance with B.S. 2960 : 1958. The use of the improved insulation makes it possible to offer a motor of smaller dimensions at a lower price than the earlier Class "A" insulated 55° C. rise British Standard Dimension machine which it replaces. The new motor is immediately available in sizes up to $7\frac{1}{2}$ h.p. but at a later date the range will be extended to 40 h.p. at 1,500 r.p.m.

Associated Electrical Industries, Ltd., Motor and Control Gear Division, Rugby.

Holmes-Retroflux Bag Filters

A further addition to the range of dust collection and control plant manufactured and marketed by the Gas Cleaning Division of W. C. Holmes & Co., Ltd., is the Holmes-Retroflux bag filter, which was developed by Standard Filterbau Gesellschaft m.b.H. of Münster and is to be manufactured under licence in the United Kingdom. This filter has numerous advantages over conventional bag filters and is particularly suitable for those applications where high dust burdens are encountered and/or dusts of an adhesive nature have to be collected. It has an efficient and continuous method of cleaning the filter bags which ensures that large volumes can be handled with constant pressure drop and high collection efficiency. An efficiency in excess of 99% for all particles, including those of sub-micron size can be guaranteed.

In operation, the dust laden air enters the header within the casing and is distributed to the inside of the filter

bags, the air and dust moving in the same direction. The tendency of the dust to settle by gravity is not impeded by air currents as the air passes through the filter bags radially and the air velocity gradually diminshes to zero at their lowest end. The dust collected on the inner surface of the filter bags is continuously dislodged by the passage of high velocity air jets through the fabric in the reverse direction to the main air stream. The dislodged dust falls by gravity to the worm conveyor or hopper below. The use of high velocity air jets eliminates the rapping or shaking mechanism and separate compartments sometimes encountered in bag filters.

The jet tubes have a narrow slot in the face which is in contact with the filter bag and are specially shaped and finished to avoid damage to the fabric. The ends of the tubes are set in hollow frames which are suspended by chains, and are supplied with high pressure air from a blower via a flexible hose. The frames operate in counterbalanced pairs when large volumes of air are being filtered ; each frame is capable of cleaning the complete length of up to thirty-six bags.

The jet tubes are mounted in pairs, one pair being positioned approximately 3 in. below and at 90° to the other, and as each jet tube is in contact with the surface of the bag over a quarter of its circumference only, the bag becomes slightly elliptical in shape as each pair of tubes travels along its length. This arrangement of the jet tubes ensures that the bag is not constrained in any way ; creases do not form in the fabric and wear is reduced to such an extent that filtering media such as glass fibre can be used successfully when handling hot flue gases.

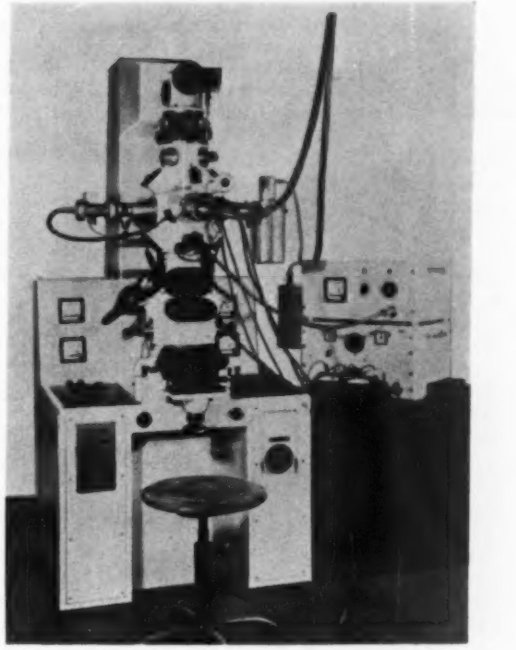
A major advantage of the Homes-Retroflux bag filter is that each bag is continuously and efficiently cleaned over its entire surface area. The high velocity jet of air which passes through the fabric is measured in thousands of ft./min. compared with less than ten ft./min. for the normal filtering air speed through the filter bag. One important result of this efficient cleaning is that the filter resistance can be maintained at approximately half of that normally experienced with a conventional bag filter without sacrificing any drop in efficiency : an appreciable saving in power consumption is thus achieved.

The Holmes-Standard bag filter, which is a combination of a conventional circular bag filter and cyclone is also to be manufactured under licence. This filter has an equally high efficiency and is recommended for less arduous duties.

Gas Cleaning Division, W. C. Holmes & Co., Ltd., Turnbridge, Huddersfield.

Electron Diffractograph KD 3

THE Trüb Täuber electron diffractograph KD3 has been designed for universal application. Problems in crystallography, physics and metallurgy can be solved using the basic apparatus with standard components. By the fitting of additional parts, the diffractograph is readily adaptable to users' special requirements. Diffraction patterns can be observed on a luminous screen or recorded on photographic plates or films, and a special motor-driven device permits the continuous recording of structural changes as a function of temperature of the object. The specimen can be etched under vacuum by ionic etching bombardment by means of a special ion gun of high current intensity with seven beams. By operating



the ion gun simultaneously with the moving film it is possible to record the progressively changing surface structural pattern as successive layers are etched away.

Comprising a tube for the electron beam with vacuum equipment and high voltage rectifier, the basic equipment permits recording of diffraction patterns from layers and surfaces, using the so-called transmission and reflection method at room temperatures. Accessories are provided for heating the object up to 900° C. and for cooling it to low temperatures. The specimen holders will move the object into any desired position and rotate it about its axis during operation without interrupting the vacuum. For direct viewing of the specimen an optical microscope is available with a magnification of 200 ×. Evaporation of metals in the diffraction chamber can be utilised to produce thin evaporated layers without exposure to the atmosphere.

The Trüb Täuber cold Induni cathode with an accelerating voltage between 20 and 50 kV., the vacuum equipment with a special oil diffusion pump, and the clear and simple arrangement of the controls make the electron diffractograph very efficient and reliable. The electron optical accessories in the apparatus make it possible to observe micro-diffraction as well as electron optical magnification of the object of about 70 ×.

Sole Agents in Great Britain for Trüb Täuber & Co., Ltd., are H. Tinsley & Co. Ltd., Wendee Hall, South Norwood, London, S.E.25.

Portable Mains-Operated Beta Gauge

BALDWIN INDUSTRIAL CONTROLS have recently introduced into their range of nucleonic thickness gauges a small portable mains-operated unit, which has been designed to meet the requirements of those sections of industry where the expense and refinements of the standard beta

gauge are not justified. The gauge is calibrated in arbitrary units proportional to weight per unit area, and the user can easily co-relate a factor for his own particular product.

For simplicity, the gauge embodies only one radioactive source and detector. The output from the detector is compared with a calibrated reference potential and the difference in signal is amplified by an extremely stable low-gain D.C. amplifier. The output signal is sufficiently powerful to operate both the built-in moving coil meter and, if required, an additional external milliamp recorder.

Although the instrument has been simplified to meet a specific demand, it is claimed that the quality and accuracy of its measurements are excellent within the limitations imposed by economics. Various measuring head mountings are available according to the application or trade requirements, and alternative radioactive sources are offered depending on the range and thickness or weight per unit area of material to be measured.

Baldwin Industrial Controls, Ltd., Dartford, Kent.

Indicating Temperature Controller

A new indicating temperature controller has been introduced by Honeywell Controls, Ltd. Designed for heating and cooling operations, it serves the double purpose of controlling and indicating temperatures in ovens, cookers, plating baths, dryers and similar industrial applications. A highly sensitive, remote bulb, liquid-filled thermal system operates a single-pole, double-throw, Honeywell micro-switch which can either open or close a circuit on temperature rise. A selection of ranges is available from $-50°$ F. to $+1,000°$ F., and centigrade scales are available if required. Both case and capillary are fully compensated for ambient temperature variations. The set point can be changed by means of a knob on the front of the case and both the indicated temperature and set point are made clearly visible by magnifying windows. The steel case has an attractive facia and is equipped with slotted mounting brackets so that the controller can be either surface mounted on a wall or flush mounted in a panel. Full details are given on specification sheet No. S1010-7.

Honeywell Controls, Ltd., Ruislip Road East, Greenford, Middlesex.

Thermocouple Data Indicator

An exceptionally useful graphic calculator giving thermocouple data has been introduced by West Instrument Corporation. Called the "thermocouple data indicator," the calculator gives millivolt values for iron/Constantan, copper/Constantan, Chromel/Alumel, platinum-13% rhodium/platinum and platinum-10% rhodium/platinum thermocouples for all temperatures within their range. It will indicate directly for readings based on a zero degree reference temperature or corrected to any ambient temperature. This makes the thermocouple data indicator especially valuable when used with potentiometer recorder calibration.

Other data given includes thermocouple and extension wire resistances; conversion of B & S wire gauge into both millimeters and decimal inches; pipe sizes, including outside diameters and wall thickness for standard, extra heavy and double extra heavy pipe; Instrument Society of America wire type designations and colour codes; maximum temperatures for common thermocouple protection tubes; thermocouple polarity; fahrenheit-centigrade conversion; and standard C and D scale slide rule.

West Instrument Corporation, 4363 West Montrose Avenue, Chicago 41, Illinois, U.S.A.

Electric Motors

A new range of totally enclosed fan-cooled motors which are smaller, lighter and cheaper than existing motors of the same type is announced by English Electric. The dimensions of the new motor are exactly the same as the "C" type ventilated motor (B.S. 2960), and the two types are, therefore, interchangeable. The new motors, called type "D" have outputs up to 100% greater than existing motors of the same size, (type "B"). The greater outputs have been achieved by allowing for a temperature rise of 65° C. above 40° C. instead of the existing 55° C. This results from the use of class "E" insulation instead of class "A".

These new motors are designed for wide industrial application, particularly where there is dust and dirt in the atmosphere. Special versions, having internal and external anti-corrosion treatment, will be produced for the chemical and gas industries. The twelve frame sizes cover ½-40 h.p. in 2-, 4-, 6- and 8-pole machines, and the motors can be wound for operation on any 2 or 3 phase supply up to 650 volts at 50 and 60 cycles. They are also available for any specific frequency such as the 25 cycle or 40 cycle supply. They will give an average starting torque of approximately 200% full load torque with an average starting current of 700% full load current when started direct-on-line. High torque motors will also be available where special starting characteristics are required.

The English Electric Co., Ltd., Marconi House, Strand, London, W.C.2.

CURRENT LITERATURE

Book Notices

HIGH PRODUCTIVITY IN HEAVY ENGINEERING

Production Inspection and Cost Control in Welded Fabrication

By A. G. Thompson. $8\frac{3}{4} \times 5\frac{1}{2}$ in., 339 pp. including 36 plates. Published for *Welding Metal Fabrication* by Iliffe & Sons, Ltd., London, 1960. 65s. net (by post 66s. 3d.)

The introduction of welded fabrication into heavy engineering has led to the development of a whole range of new techniques. These techniques have revolutionised the production methods of many firms and, indeed, of complete industries, of which shipbuilding is an outstanding example. In addition, the development of the modern oil industry and commercial nuclear power has been largely dependent on the new techniques, which also find application in the manufacture of pressure vessels, liquid storage containers, machine and engine frames, and in structural engineering.

This book has, as far as possible, avoided repeating what has already been published on welding. In the main it is about the technology which has been built up round welded fabrication; accurate dimensioning, coding methods of transferring dimensions, cutting machines—including computer-controlled flame profiling machines, using punched or magnetic tape—and welding machines. A very full chapter deals with the latest metal-working processes essential for efficient fabrication, and includes details of the various machines which have been specially evolved for pre-forming sheet and plate. Other chapters deal with shop layout; assembly and handling; and quality control and inspection, where information is given on the latest methods of non-destructive testing.

The second half of the book is devoted to costing methods appropriate to heavy fabrication. The four chapters on various aspects of this subject include one on productivity improvement, which illustrates, by examples taken from heavy engineering, the specialised approach which is necessary.

This well illustrated book, by an industrial consultant who has made a particular study of this highly specialised subject, will be invaluable to all those responsible for the fabrication of metal by welding—management, engineers, designers, production and planning staffs; while students of all branches of engineering should also find much to interest them.

MODERNISATION OF FORGES

Proceedings of conference on modernisation of forges organised by the British Iron and Steel Research Association in September 1958. 31 pp., photographs and diagrams. Obtainable from the Association's Information Distribution Officer, 11 Park Lane, London, W.1. Free to representatives of B.I.S.R.A. member firms; 10s. post-free to non-members.

The full proceedings of the conference on modernisation of forges, sponsored by the Mechanical Working Division of the British Iron and Steel Research Association, and held at Ashorne Hill in September 1958, have now been made generally available. In opening the conference the chairman, Mr. F. J. Somers, said that those who had been fortunate enough to visit recently forge plants in America and Germany would have been impressed by the improvements in methods in those countries. One of the main objects of the conference was to spark off an exchange of ideas which would eventually be translated into action in the various forge shops in this country, with the object of ensuring that the forging industry in Britain was at least abreast of, and if possible ahead of, developments elsewhere.

Three papers were presented as a basis for discussion. In the first of these, Mr. A. C. Lowe (Thos. Firth & John Brown, Ltd.), related some of their experiences in building a new forge as part of their general forge development scheme. The forge concerned is equipped with a 1,750 ton air-hydraulic press, bogie hearth furnaces, and a rail type manipulator. Mr. E. H. Kendall (Kendall Contracting Inc., U.S.A.) then discussed the features that make for a well-designed modern forge. Breaking down the forging operation into its successive stages, Mr. Kendall followed a consideration of forge layout with sections dealing with reheating furnaces; presses, hammers, and manipulators; and cooling and heat treatment facilities. The aim of the final paper, by Messrs. J. Banbury, I. V. Chelson and D. H. Smellie (B.I.S.R.A.) was to outline a method of comparing different forging techniques; to show how the method had been used for a company to compare an existing forge with a proposed new forge; and to show how it was being used to make comparisons between companies. The full texts of the papers are included in this publication, together with a record of the subsequent discussion.

Trade Publications

A grey iron casting forming part of a water pump component suffered from porosity in a heavy section beneath the riser. This fault persisted in spite of an attempt to cure the trouble by using two risers instead of one. An account is given in *Foseco Foundry Practice* No. 138 of an investigation of the problem carried out by the service personnel of Foundry Services, Ltd., Long Acre, Nechells, Birmingham, 7, from whom copies may be obtained. Also featured in this issue are a section dealing with grain refinement of aluminium alloys, and a forecast, written in the style of the prophets of old, of conditions in the foundry industry in 1978.

"Chemicals for research and analysis" is the title of an illustrated leaflet issued by Hopkin and Williams, Ltd., outlining the part the company plays in producing chemicals to AnalaR standards for use in chemical research and analysis. Reference is also made to the fact that the company also publishes individual monographs on selected reagents and reference books on certain ranges of chemicals and reagents.

Heat is essential at some stage or another in the manufacture of the almost endless variety of articles we use in everyday life. Heat is called for in the melting of non-ferrous metals, in hardening, annealing and tempering of metals, in metal spraying and stove enamelling, in the manufacture of motor vehicles, in paint and varnish boiling and drying, in type casting, in glass blowing, in pottery production, in baking and confectionery, in the electrical industry, in shipbuilding and many many more industries. In a new leaflet recently issued by the Gas

Council, are given examples of what one therm of gas will do in these various activities.

The term " pressure vessel " is wide-embracing and can be applied to components varying from fabrications in mild steel plate or sheet, to massive forgings in alloy steels, the mode of construction being dependent upon operational conditions. Because of the accumulated experience in the manufacture of pressure vessels and the manufacturing facilities available, the Hadfields Group of Companies is able to supply vessels made as forgings, steel castings, fabrications, iron castings, centrifugally cast steel, iron or non-ferrous metal, or a combination of these methods. A new booklet, No. 544, issued by the Hadfields Group, discusses pressure vessels made in these various ways and illustrates typical examples. Reference is also made to the metallurgical and design facilities available.

One of the most important factors in successful anodising is the jigging of the articles or components being treated. Repairs to jigs, re-cycling of articles imperfectly anodised because of damaged contacts, and loss of throughput arising from faulty or broken tips, all make their contribution to the cost of anodising—and an appreciable contribution it may be. The main difficulty has been the short life of the jig materials, but with the advent of titanium it is now worth while to invest in well designed and carefully constructed jigs. It is estimated that the extra first cost of titanium jigs is recovered in the first few weeks of operation, and a new leaflet—No. 8 in the series " I.C.I. Titanium for Chemical Plant "—issued by I.C.I. Metals Division, discusses the production and use of all-titanium anodising jigs for long runs and titanium-tipped jigs for short runs.

Over the past few years, processes of pneumatic steelmaking employing commercially pure oxygen produced in " tonnage " plants have had a revolutionary effect on the world's steel industry. The probability of their increasing influence in the future and the extent to which they should immediately replace the existing steelmaking methods are topics of prime importance in industry today. The impact of pneumatic steelmaking has been so great that a considerable volume of technical literature, much of it stressing the virtues of individual processes, has already accumulated. The author of an article in *Murex Review*, Vol. II, No. 21, attempts to describe these processes in simple terms for the non-specialist reader, to clarify their relationship with each other and to trace their development from the original Bessemer process.

The growth of Ashmore, Benson, Pease and Co., Ltd., from a small jobbing engineering works to an engineering organisation offering a complete service to the heavy industries of the world, made essential a programme of reconstruction of the manufacturing facilities after the end of the last war. The products of the company have grown too large and too heavy to be economically manufactured in the old shops, and it was decided to construct a new factory on a site about a mile away from the original Parkfield Works. A copiously illustrated brochure has recently been issued, illustrating the new facilities available, including a constructional shop, machine and fitting shop, and a stockyard for plates and sections. The foundry, on an adjacent site, is capable of producing 5,000 tons of castings a year in grey iron, Meehanite and spheroidal graphite iron, up to 25 tons in weight. Apart from manufacturing facilities, there are sections dealing with inspection and materials testing, transportation—which is a major problem with these large constructions—and site erection. The final section illustrates the wide range of blast furnace plant and equipment manufactured by the company.

The December issue of *The English Electric Journal* has a distinctly metallurgical flavour. The article on the estimation of fatigue life of aircraft structures is admittedly mainly concerned with the engineering aspect, but the remaining articles are all concerned with electrical plant for the metal industries. They include electrical controls in the steel industry, mercury arc convectors for rolling mill duties, and tinplate flow-melt equipment at the Velindre Works of the Steel Company of Wales, whilst the double-page spread coloured illustration is of a 5,000 h.p. drive for a hot reversing plate mill.

Books Received

" Modern Methods of Analysis of Copper and its Alloys." By C. M. Dozinol. English translation by G. R. Andraso. 239 pp. Brussels, 1960. Ets. George Thone. $32.

" Nuclear Fuel Elements." Edited by H. H. Hausner and J. F. Schumar. 409 pp. inc. index. New York and London, 1959. Reinhold Publishing Corporation and Chapman & Hall, Ltd. 100s. net.

" Liquid-Metal Heat Transfer Media." By S. S. Kutateladze, V. M. Borishanskii, I. I. Novikov and O. S. Fedynskii. Translated from Russian. (Supplement No. 2 of the Soviet Journal of Atomic Energy, *Atomnaia Energiia*. 149 pp. New York and London, 1959. Consultants Bureau, Inc., and Chapman & Hall, Ltd. 180s. net.

" Fracture." Proceedings of an International Conference on the Atomic Mechanism of Fracture held at Swampscott, Massachusetts, 12th to 16th April, 1959. Editors : B. L. Averbach, D. K. Felbeck, G. T. Hahn and D. A. Thomas. 646 pp. inc. index. Published jointly by The Technology Press of Massachusetts Institute of Technology and John Wiley & Sons, Inc., New York, 1959. Chapman & Hall, Ltd., London. 140s. net.

" Radioactive Tracers in Chemistry and Industry." By P. Daudel. Foreword by Irène Joliot-Curie. Text augmented and revised by the author. Translated by U. Eisner. 210 pp. inc. index. London, 1960. Charles Griffin & Co., Ltd. 36s. net ; by post 1s. extra.

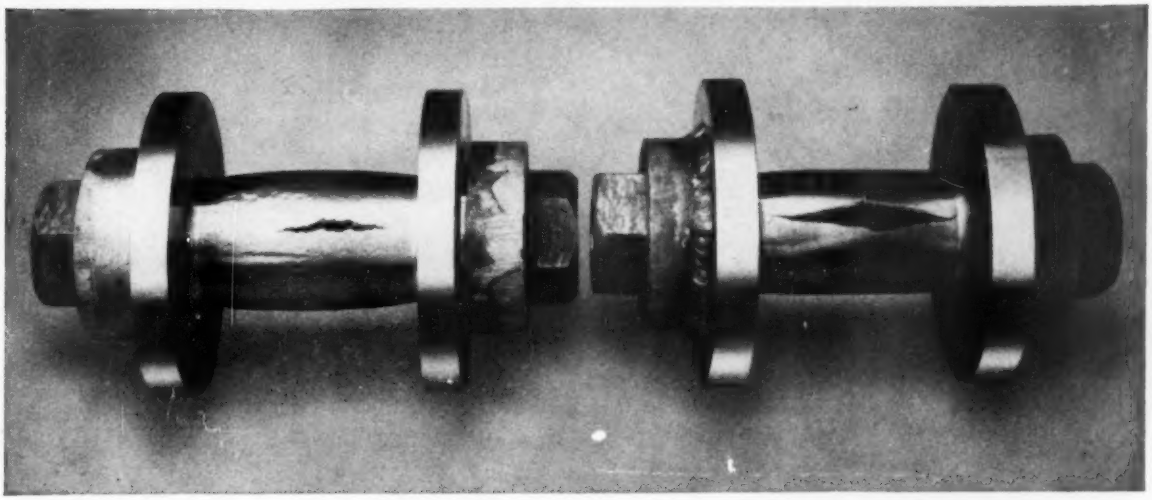
" Physical Metallurgy." By B. Chalmers. 468 pp. inc. index. New York and London, 1959. John Wiley & Sons, Inc., and Chapman & Hall, Ltd. 100s. net.

" General Crystallography." By W. F. de Jong. 281 pp. inc. name and subject indexes, and numerous illustrations. San Francisco and London, 1959. W. H. Freeman and Company. 38s.

" Electronics for Spectroscopists." By members of the Photoelectric Spectrometry Group and of the Electronics Department of Southampton University. Edited by C. G. Cannon. Schwarz Memorial Volume. 333 pp. inc. index. London, 1960. Hilger & Watts, Ltd. 60s.

REMARKABLE PERFORMANCE OF ALUMINIUM BRONZE

0·10 IN. SHELL WITHSTANDS OVER 2 TONS PER SQ. IN. !



Recently we tested to destruction two similar hollow castings—one in gun-metal, the other in aluminium-bronze. The gun-metal had a wall thickness of **0·25 in.** and withstood pressures up to 4480 lbs. p.s.i.—a very good performance. The casting in aluminium-bronze burst at a slightly higher pressure (4816 lbs. p.s.i.) **but its wall thickness was only 0·10 in.** Surely an impressive demonstration of the weight/strength characteristics of this alloy.

We were one of the first foundries to cast in aluminium-bronze and to-day we supply castings in this alloy to customers all over the world.

LEFT *Gun-metal* **0·25 in.** *thick—burst at 4480 lbs. per sq. in.*

RIGHT *Aluminium-bronze* **0·10 in.** *thick—burst at 4816 lbs. per sq. in.*

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in all non-ferrous alloys. Precision machining. Specialists in aluminium-bronze, centrifugal-cast wheel blanks, shell moulded castings, and chill-cast rods and tubes. Continuous cast phosphor bronze bars up to 12 ft. lengths.

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B.E.R.L

LABORATORY METHODS

MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC

INSTRUMENTS AND MATERIALS

APRIL, 1960. Vol. LXI, No. 366

The Analysis of Nickel

Part II. The Application of a Spectrographic Solution Technique to the Analysis of Nickel

By G. P. Mitchell and G. Orme

Materials Research Laboratory, The Mullard Radio Valve Co., Ltd.

Part II of this paper is concerned with two spectrographic methods of analysis for electronic type nickel alloys. The first method, a powder technique due to E. K. Jaycox, is discussed only from the point of view of modifications which the present authors consider necessary for routine practice. The second technique, a direct solution one, is made possible by the use of a new solution electrode. A description is given of this electrode and some general properties to be expected when used in solution analysis. Finally, a method of nickel alloy analysis, together with some typical results is reported.

IN Part I of the paper, by T. R. Andrew and C. H. R. Gentry*, a description is given of the chemical procedures devised by them for the determination of the elements which are present as alloying or impurity elements in "electronic" type nickels. Whilst the procedures are suitable for rapid routine determinations, the logical course, if routine analysis on a regular scale is contemplated, is to employ a spectrograph. The analytical requirements which are to be met by spectrographic analysis are given in Table I.

The choice of any spectrographic procedure must be carefully considered in the light of factors which may impose limitations on the type of procedure employable. The electronics industry receives or fabricates nickel into many shapes and sizes: thus, nickel sheet, tubing, wire and powder are often submitted for analysis. Apart from the physical form of the sample received, the amount of sample is of importance, since a quantitative analysis of a single valve cathode weighing a few milligrams is sometimes required. These limitations necessitate a technique of analysis capable of handling all types of nickel sample, preferably by reducing to a common physical form, such as a powder, a solution, or metal filings. Two further conflicting factors, the question of accuracy and range of element determinations, limit one's choice of technique. From accuracy considerations the natural choice is a spark type of excitation, yet the ranges quoted in Table I require a D.C. arc type of excitation to attain the quantitative sensitivity required. The vexed question of the availability of "standards" still remains. Hitherto, no commercially available nickel standard alloys were obtainable, consequently the spectrographic technique is limited to one which can use synthetic standards.

From the above considerations it is clear that the choice of spectrographic technique is limited, for nickel analysis, to a powder or solution method with an excitation of the "arc" type capable of giving high quantitative sensitivity and accuracy. It is the intention in this paper, therefore, to discuss a powder technique of nickel analysis and also to describe a new solution technique, which it is believed may be generally used for materials other than nickel.

TABLE I.

Element	Range (%)
Magnesium	0·001–0·20
Cobalt	0·01 –1·0
Copper	0·005–0·30
Manganese	0·001–0·50
Iron	0·005–0·50
Titanium	0·005–0·10
Aluminium	0·005–0·20
Chromium	0·001–0·20
Silicon	0·001–0·50

A POWDER D.C. ARC METHOD OF NICKEL ANALYSIS

Relatively few spectrographic papers have been published on nickel analysis. Keeping in mind the limitations for the authors' purpose, most of these methods[1-5] have been examined as to their suitability in routine practice. Of these, the most useful procedure is that suggested by E. K. Jaycox[5] and subsequently put forward as a tentative method of nickel analysis by the American Society for Testing Materials.[6] The method is fully described in the A.S.T.M. publication, and the reader may refer to this for details.

This method has been adopted in essentials and used satisfactorily in these laboratories over a period of ten years. However, the authors would like to offer here one or two practical modifications, which, although having no significant influence on the reported accuracy of deter-

* METALLURGIA; **60**, 27–30, 69–72, 121–124, 173–176.

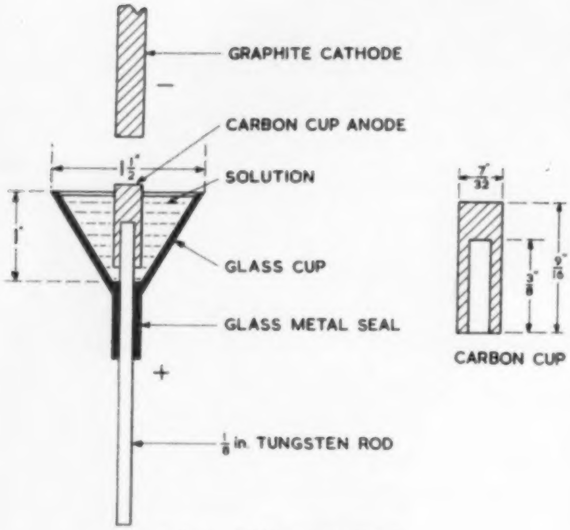

Fig. 1—Solution electrode.

minations, do improve the reliability of the method in routine practice.

(1) The history of the sample prior to actual analysis may influence the analytical result; e.g., there may be contamination either by handling or processing. It is advisable, therefore, to adopt a standard cleaning technique for all samples before taking into solution with nitric acid. A suggested cleaning technique will be described later in the paper.

(2) When many nickel samples are received for analysis each day, the provision of platinum ware for the evaporation process becomes a costly exercise. It has been found quite satisfactory to use silica crucibles, with the proviso that they should be rejected if the glazing of the interior surface is at all suspect after repeated use.

(3) The authors' experience showed that after drying at 400° C. the sample had a pronounced tendency to stick to the sides of the vessel in which it had been evaporated to dryness. It was found much better to add the required amount of carbon buffer to the sample during solution, evaporating the whole to dryness, then heating at 400° C., when a carbon-sample cake is produced, easy to remove from the crucible and needing only one grinding operation to be ready for arcing. This technique also reduced the risk of accidental contamination during grinding operations.

(4) Although the method specifies a sample weight of 50 mg., it is possible with only slight loss in accuracy to work with sample weights down to 5 mg., provided only duplicate spectra are recorded.

A disadvantage of the Jaycox method, from the authors' point of view, is the relatively long period of time required for the preparation of samples prior to spectrographing. This becomes more serious as the number of samples submitted for analysis increases. As a more rapid sample preparation technique was necessary, the simplest course seemed to be direct spectrography of a solution of the sample. With this idea in mind, a direct solution method of analysis for these nickel alloys was investigated and is the main subject of this paper. Nevertheless, the Jaycox procedure is of necessity still used for sample weights less than approximately 100 mg.

A SOLUTION TECHNIQUE FOR NICKEL ANALYSIS

The choice of a direct solution technique for nickel analysis was primarily determined by the need to reduce the time factor in sample preparation and to be independent of chemical standards. Whilst the advantages of using solutions for spectrographing are obvious, there is also a serious disadvantage. Thus, they are not easily transportable into an analysis gap, consequently there is an inherent lack of sensitivity of detection of elements in solution when excited by the normal electrical discharges. To overcome this difficulty, many workers use solutions as an intermediary in converting samples to powders, which are subsequently analysed on carbon electrodes. Others have preferred to impregnate carbon electrodes with solution, which are then dried and arced or sparked in the usual way. The passage of a spark discharge from a solid electrode to a solution supported in a cup type electrode is a technique described by Gerlach and Schweitzer[7] in the early days of spectrographic analysis. Beal and Poynter[8] have used a more refined type of copper cup electrode for solution analysis, whilst very recently, Ho I-Djen and Li Shih-Cho[9] have described a cup electrode capable of being used with an A.C. type of arc discharge carrying an average current of approximately 6 A. This is a very attractive feature of the electrode, since the immediate possibility exists of increasing the sensitivity of analysis of elements in solution. Nevertheless, there were some objections to this electrode when used in routine practice, and in efforts to overcome them, F. Farrell and the present authors used a new cup type electrode described in a note submitted to *Spectrochimica Acta*.[10]

Apparatus

Cup Electrode

In Fig. 1 is shown the construction of the cup electrode, together with the dimensions of the carbon cap which is fitted on to the tungsten post. In spite of the glass-metal seal, experience has shown this electrode to be quite robust in operation, and able to withstand relatively high average currents from multisource discharges over comparatively long periods of time. It is very resistant to chemical attack, so that either acidic or basic solutions may be used if so desired. One real advantage of the electrode is that, since the discharge does not occur directly on to the solution surface, sputtering of solution droplets to the detriment of the surrounding apparatus is not evident.

It is important to see that the carbon cap is a tight push fit on the tungsten post, otherwise, during the passage of a discharge, a loose cap will be ejected into the analysis gap. For this reason it is advisable to maintain a stock of carbon caps for one particular cup electrode, and use only that electrode for a series of exposures.

In use, the cup electrode is fitted with the carbon cap which protrudes 1 mm. above the rim of the glass cup. A simple 1 mm. spacer may be devised for this purpose, whereby the correct distance will be maintained from exposure to exposure. The electrode is placed in the arc

stand and connected to the positive side of the source unit. The cathode, either pure graphite or carbon, is placed in the upper position in the stand with an analysis gap setting of 3 mm. The solution to be analysed is pipetted into the cup until level with the rim ; the discharge may then be started.

Unlike the Ho I-Djen electrode, where solution is fed into the analysis gap by seepage over the surface of the small copper anode, the present electrode utilises the wick action of the carbon cap, this being more reliable, especially if the level of solution in the reservoir changes through evaporation, as is the case where high energy discharges are being used.

Spectrograph

A fully automatic large Hilger spectrograph, having ample dispersion for nickel alloys, was used on all the work described in this paper.

Excitation Equipment

A Hilger-B.N.F. multisource unit capable of giving D.C. discharges up to 10 A., and, by suitable choice of the electrical parameters, a diversity of discharges of the overdamped type, differing only in the " burning time " and peak current of the discharge, may be produced.

Microphotometer

A Hilger non-recording type microphotometer with plate motor drive was used in conjunction with a galvo-scale projector.

Calculating Board

The equipment employed for all calculating requirements was a commercial Respektra board.[11] A multiplicity of calculating operations may be expediently performed with this equipment, and it has been found to be especially useful where background corrections have to be made.

Developing Equipment

A commercial A.R.L.-Dietert developing machine was used for all plate processing operations, the equipment being maintained at a temperature of 18° C. by thermostat.

Preliminary Experiments

Before the final technique for the spectrographic analysis of nickel could be put into practice, many preliminary experiments had to be made to ascertain the performance of the cup electrode. It is impossible to report all of these, but mention will be made of some of the more important facts which apply not only to nickel analysis but to solution analysis in general.

Size of Cup Electrode

The size of the reservoir of the cup electrode is dependent upon two factors—the type of discharge used, and the strength of solution to be analysed. With the spark type of discharge such as that given by the Hilger spark set, very little solution is lost by feed-through to the analysis gap, consequently the cup may be of such a size that only 2 ml. of solution are required. As one goes to the higher energy discharges, as given by multisource units, more of the solution is lost, both by volatilisation in the analysis gap and evaporation through heat dissipation in the solution. In this case the cup must be capable of holding 5–7 ml. of solution, suitable dimensions for this volume being given in Fig. 1. Apart from the above consideration, a compromise is necessary in the choice of solution strength to be used for analysis. For nickel, the choice was 10 mg./ml., which, with a cup holding 5 ml. of solution, requires a minimum sample weight of 100 mg. for a duplicate analysis. Because of the low solute-to-solvent ratio a long exposure period is required for satisfactory spectra. In this way maximum loss of solution occurs, and this gives rise to the necessity for having a 5–7 ml. cup electrode. From purely spectrographic considerations, it would be better to use a high solute-to-solvent ratio, say, 40 mg./ml., which would require a shorter exposure period and a smaller volume cup, since less solution would be lost. However, two or three times the sample weight would be required for an analysis—as compared with the authors' choice—which is not acceptable having regard to our previously stated requirements for nickel analysis.

Sensitivity of Detection

Having ascertained the most suitable physical dimensions for the electrode under the various excitation conditions, a most important question was that of sensitivity of detection with the electrode. Referring to Table I, it will be seen that the required absolute sensitivity of detection in solution of the elements quoted, is of the order of 0·1 p.p.m. on the basis of 10 mg. of nickel in 1 ml. of solution.

Experiments were therefore undertaken to determine the most suitable excitation conditions. It was soon apparent that only the most powerful multisource discharge could attain such a sensitivity of detection. The electrical parameters of the most suitable discharge were : $C = 250\,\mu F.$; $R = 15\,\Omega$; and $L = 0{\cdot}06$ mH. ; as shown on a Hilger-BNF multisource. The average current of this arc-like discharge is of the order 6 A., and is overdamped.

Acid solutions of the elements present in nickel (10% hydrochloric acid was preferred whenever possible, but nitric or sulphuric solutions were also used if necessary) were made up in the proportions of 1,000, 100, 10, 1 and 0·1 p.p.m. Using the arcing conditions just stated, duplicate spectra were obtained for each solution, and from these spectra approximations were made of the sensitivities of detection for each element. A comparison was then made with the results for sensitivities quoted by C. Feldman.[12] Encouraged by these results, the work was extended to many other elements not present in nickel, the whole being reported in Table II.

It is clear from these results that the cup electrode offers a generally increased sensitivity over the porous cup electrode of Feldman. Whilst the increase in sensitivity is not outstanding in the case of the first nine elements quoted in Table II, i.e. those elements present in nickel, it is of a sufficient order to cover most of the lower limits of the ranges quoted in Table I. Some of the figures quoted in Table II for sensitivity are modified to some extent by the presence of some elements as impurities in the electrodes. Two notable cases of this are silicon and magnesium, where the probable lower limit is signified as less than 0·1 p.p.m.

The characteristics of the spectra obtained under the stated conditions also influence the choice and sensitivity of lines available for analytical purposes. Thus, where spark-like discharges are used, solution spectra are obtained relatively free from background, cyanogen

TABLE II.—SENSITIVITIES OF ELEMENTS IN SOLUTION USING A CUP ELECTRODE

Element	Line Used	Sensitivity (p.p.m.)	
		Feldman Porous Cup Electrode	Authors' Cup Electrode
Aluminium	3961·527	1·0	0·1
Copper	3247·540	0·6	0·1
Iron	2599·396	2·5	0·5
Chromium	2843·252	2·0	0·1
Cobalt	3453·505	2·0	0·5
Manganese	2593·729	2·0	0·1
Magnesium	2795·53	0·01	<0·1
Titanium	3349·035	3·0	0·5
Silicon	2881·578	—	<1·0
Nickel	3414·765	10·0	0·5
Silver	3280·683	1·0	0·1
Boron	2497·733	0·5	0·5
Barium	4130·664	50·0	10·0
Calcium	3968·5	—	0·1
Beryllium	3131·072	0·02	<0·1
Bismuth	3067·716	5·0	0·5
Mercury	2536·519	50·0	1·0
Indium	3256·090	10·0	1·0
Potassium	4044·140	200·0	100·0
Sodium	3302·323	35·0	10·0
Lead	2533·069	10·0	1·0
Antimony	2598·062	100·0	5·0
Tin	2839·989	100·0	1·0
Strontium	4077·714	0·5	0·5
Vanadium	3093·108	5·0	1·0
Tungsten	4008·753	500·0	100·0
Zinc	3345·02	25·0	10·0

bands, and band structure associated with the use of solutions. Under the quoted conditions of the multi-source discharge, spectra are characterised by their arc-like nature and relative freedom from background up to about 3,000 Å. At wavelengths higher than this, band structure is very evident; not only are the cyanogen bands present, but other band structure, probably due to hydroxyl radical, may be seen, a fact which was observed by Feldman.[12] Most of his other observations regarding solution spectra are also applicable to the authors' solution electrode. This band structure interferes with the sensitive zinc lines in the 3,340 Å region, a decided draw-back, since they are the most sensitive ones available to the spectrographer. Quantitative determinations using the aluminium 3092 Å lines were also erratic due to interference from weak band lines. It is advisable, therefore, where quantitative determinations are to be made, to examine carefully all lines used above 3100 Å for this possible type of interference.

Table II quotes a sensitivity figure for silicon, and it was also noticed, during preliminary experiments with potassium silicate solutions, that quantitative silicon estimations should be possible. Further work confirmed that there existed no real obstacle to the determination of silicon in nickel solutions, a finding which is incorporated in the final technique.

Effect of Tungsten

Tungsten may be present as an alloying constituent up to 4% in certain nickel alloys. Various complexing agents were tried to keep the tungsten in solution, the best of which appeared to be phosphoric acid. However, this reagent was not entirely satisfactory, and tests were then conducted to observe the effect of leaving the tungstic oxide in suspension. Accordingly, tungsten-containing alloys were dissolved in nitric acid and analysed using the cup electrode: (*a*) with the tungstic oxide in suspension; and (*b*) with the tungstic oxide filtered off. Results obtained on two samples are shown in Table III, from which it will be seen that tungstic oxide in suspension does not affect the analytical result, although, surprisingly, the oxide enters the discharge gap, as evidenced by the presence of tungsten lines in the spectra. The particles must therefore be transferred through the carbon cap, but no attempts have been made to ascertain whether this transfer is quantitative or not.

Final Analytical Method

Sample Preparation

Before analysis of a received sample is attempted, a standard cleaning procedure is adopted. The sample is first degreased in trichlorethylene, or any suitable degreasing solvent, and dried. Approximately 20 ml. of 1:1 hydrochloric acid is then placed in a small Pyrex beaker and brought to the boil on a hotplate. A 0·5 g. sample is placed in the acid and boiling is continued for a 1½ minute period. The beaker is then removed from the hotplate and rapidly cooled by adding distilled water. Washing is continued, with the sample still in the beaker, until no trace of hydrochloric acid can possibly be left. To the sample, 10 ml. of 1 : 1 nitric acid is added and the beaker warmed gently until solution is complete, final boiling driving off brown fumes and leaving a clear solution. If tungstic oxide is present, it is left as a suspension. The nickel solution is transferred to a 50 ml. standard flask and made up to the mark with distilled water. This solution is then used for analysis. Where a sample weighing 0·5—0·1 g. is submitted, the procedure is scaled down, but below 0·1 g. of sample the Jaycox method is used.

Standard Preparation

Synthetic standards are used for this method, being prepared either from pure metals or salts of metals, whichever is more convenient. The synthetic standards are prepared from the following standard solutions:—

Nickel.—The matrix nickel solution is prepared from a chemically analysed Mond nickel powder. Sufficient powder is dissolved in 1:1 nitric acid (1 g. of powder requires 20 ml. of the acid) to make a 10 mg./ml. stock nickel solution to cover all requirements for synthetic standard preparation.

Aluminium.—A standard solution—20 μg./ml. is suitable—is made in the following manner: The required amount of pure aluminium foil is dissolved in hydrochloric acid and evaporated to dryness. The residue is dissolved in the minimum of dilute nitric acid and made up to the required solution strength.

Titanium.—Pure titanium powder is dissolved in 1:8 sulphuric acid; the titanium is then precipitated as hydroxide, filtered and dissolved in the minimum amount of 1:1 nitric acid.

Silicon.—Potassium silicate may be obtained commercially in a high state of purity. It is thus only necessary to dilute an assayed solution to give the required standard solution.

Chromium.—A standard solution of potassium dichromate may be used.

TABLE III.—EFFECT OF TUNGSTIC OXIDE SUSPENSION

Sample	Location of Tungstic Oxide	Analytical Results						
		Fe (%)	Mg (%)	Si (%)	Co (%)	Cu (%)	Mn (%)	Cr (%)
A	In suspension	0·06	0·009	0·04	0·042	0·016	0·007	0·018
	Filtered off	0·05	0·010	0·035	0·043	0·018	0·007	0·020
B	In suspension	0·07	0·039	0·038	0·04	0·02	0·005	0·035
	Filtered off	0·078	0·049	0·044	0·046	0·023	0·007	0·035

Manganese.—Potassium permanganate is most suitable as a standard.

Magnesium, Copper, Iron and Cobalt.—These may be conveniently prepared as solutions from the commercially obtainable pure metals.

Having obtained solutions of the impurity metals of known strength, it is a simple matter to prepare a series of synthetic nickel standards, simply by addition of a known amount of each impurity solution in turn, to the standard nickel matrix solution. The impurities, should of course, cover the impurity ranges of Table I. The authors consider that synthetic silicon standards are more conveniently prepared freshly, as required, since no reliance can be placed on standard silicon solutions which have been standing for some time.

Spectrographic Conditions

Electrodes.—The upper cathode electrode is a 6 mm. diameter, flat-ended graphite rod—Johnson Matthey type J.M.4B may be used. The lower electrode, the anode, is the cup electrodes previously described (see Fig. 1). The analytical gap setting between the electrodes is initially 3 mm., and no attempt is made during the exposure period to maintain this distance.

Excitation.—The Hilger–BNF multisource unit settings are: capacity 250 μF; resistance 15 Ω and inductance 0·06 mH.; and the average current is approximately 6 A. The discharge is checked for stability on an oscilloscope, and the phasing control used, if necessary.

Exposure Conditions.—The construction of working graphs, using the synthetic standards, is undertaken whenever a new batch of photographic plates or developer is used. Duplicate spectra of samples and standards are photographed and the average log intensity ratio is taken for one analytical result. When the electrodes are placed in position, solution is pipetted into the cup until level with the rim. The discharge is started and a preburn period of 15 seconds is given. The spectrograph slit is then opened, and an exposure of 150 seconds duration is made. The solution is tipped out of the electrode into a beaker and the carbon cap removed. The electrode is then washed with distilled water from a chemical wash bottle and the tungsten post is dried with filter paper. A new carbon cap is fitted using a spacer to maintain the cap 1 mm. above the lip of the cup. The cup is replaced in the stand and is ready for the next operation.

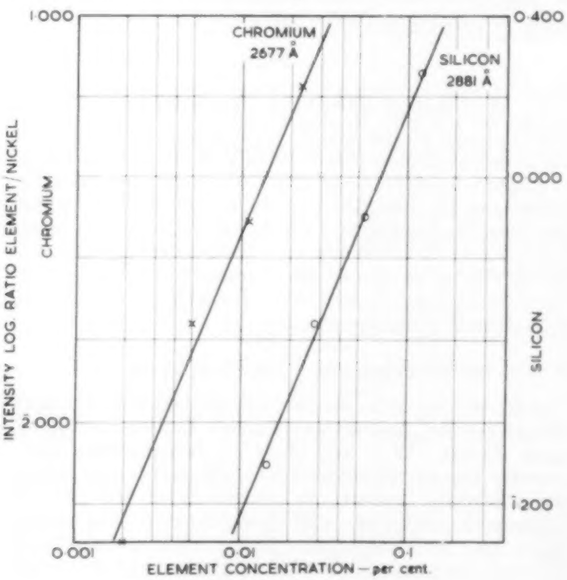

Fig. 2—Typical working curves.

TABLE IV.—ANALYSIS LINES USED IN NICKEL ALLOYS

Element		Analytical Line	Internal Standard	Concentration Range (%)
Silicon		2881·58	Ni I 2834·55	0·01 –0·40
Aluminium	(a)	3961·53	2834·55	0·005–0·10
Iron		2599·39	2834·55	0·005–0·50
Copper	(a)	3273·96	2834·55	0·001–0·50
Magnesium		2779·83	2834·55	0·01 –0·30
	(a)	2852·13	2834·55	0·001–0·02
Manganese		2933·06	2834·55	0·01 –0·50
		2593·73	2834·55	0·001–0·10
Chromium		2677·17	2834·55	0·005–0·10
Titanium	(a)	3383·77	Ni I 3232·96	0·003–0·10
	(a)	3349·40	3232·96	0·005–0·10
Cobalt		3044·00	Ni I 2834·55	0·005–1·00

(a) denotes that the 12½% transmission step of the spectra is photometered.

Spectrograph Settings.—

Spectral range	for all elements other than aluminium 2450–3500Å. for aluminium determination 2690–4400Å.
Slit width	20μ.
Slit height	2 mm.
Step sector	A revolving step sector is placed immediately before the slit, to give 100% and 12½% transmission steps.
Source to slit distance . .	The source is placed 38 cm. from the slit.

Photographic Conditions.—The plate type used is the Ilford Zenith. This emulsion is somewhat faster with a lower contrast factor than the usual types employed for quantitative work. No disadvantage has been experienced due to these characteristics, but if analytical sensitivity is not of primary importance the Ilford ordinary plate may be used also. The Zenith plate is developed in Ilford ID 2 developer for 90 seconds at 18° C., fixed in an acid hardener fixer, washed and dried.

Photometry.—A seven step filter—ratio of steps 1:2—is used with an iron arc source to calibrate the photographic emulsion. The density of the Fe line 2699·11 Å is measured, and using a Seidel[13] function, the graph relating intensity to blackening of a photographic plate is drawn on the Respektra Board. Table IV gives the wavelengths of the impurity lines measured in the nickel alloys with their percentage ranges.

A representative plot of log impurity concentration versus log intensity values, is shown for synthetic chromium standards and for synthetic silicon standards in Fig. 2.

Results

The work described here was carried out in two separate spectrographic laboratories within the same organisation. In order to check the technique, advantage was taken of a set of Mond Nickel Company spectrographic sub-standards kindly provided by Henry Wiggin & Co., Ltd. These standards had a greater

TABLE V.—COMPARISON OF CHEMICAL AND SPECTROGRAPHIC ANALYSIS OF STANDARD NICKEL SAMPLES

Sample No.	Laboratory	Si (%)	Mg (%)	Mn (%)	Cu (%)	Fe (%)	Co (%)	Al (%)	Cr (%)	Ti (%)
A	Cert. Value*	0·235	0·213	0·018	0·014	0·50	0·055	0·009	0·010	0·01
	Spectro. Lab. 1.	0·25	0·21	0·016	0·013	0·48	0·046	0·009	0·008	0·010
	Spectro. Lab. 2.	0·25	0·23	0·018	0·014	0·46	0·041	0·009	0·009	0·013
B	Cert. Value*	0·42	0·107	0·027	0·023	0·25	0·28	0·013	0·015	0·015
	Spectro. Lab. 1.	0·33	0·11	0·025	0·024	0·21	0·26	0·012	0·016	0·013
	Spectro. Lab. 2.	0·31	0·11	0·026	0·023	0·25	0·23	0·012	0·016	0·027
C	Cert. Value*	0·18	0·049	0·058	0·053	0·09	1·04	0·019	0·024	0·026
	Spectro. Lab. 1.	0·20	0·046	0·052	0·051	0·082	0·98	0·018	0·027	0·025
	Spectro. Lab. 2.	0·20	0·044	0·032	0·055	0·11	0·94	0·020	0·028	0·027
D	Cert. Value*	0·145	0·023	0·112	0·054	0·057	0·58	0·05	0·049	0·044
	Spectro. Lab. 1.	0·15	0·022	0·11	0·052	0·054	0·56	0·04	0·047	0·044
	Spectro. Lab. 2.	0·12	0·025	0·11	0·055	0·047	0·58	0·039	0·056	0·038
E	Cert. Value*	0·095	0·015	0·24	0·44	0·034	0·14	0·070	0·083	0·080
	Spectro. Lab. 1.	0·10	0·013	0·24	0·42	0·032	0·13	0·066	0·082	0·078
	Spectro. Lab. 2.	0·12	0·015	0·28	0·43	0·029	0·13	0·069	0·083	0·072
F	Cert. Value*	0·015	0·009	0·47	0·23	0·049	0·037	0·094	0·125	0·112
	Spectro. Lab. 1.	0·010	0·005	0·51	0·23	0·037	0·022	0·091	0·12	0·11
	Spectro. Lab. 2.	0·015	0·008	0·50	0·21	0·042	0·024	0·096	0·135	0·120

* Chemical analysis.

range of impurities and a greater concentration range for each impurity than any other samples hitherto available to the authors. They were therefore analysed in both laboratories by the solution technique described and the results are given in Table V, together with certificated values for comparison.

The agreement between the two laboratories is on the whole entirely satisfactory, the analytical accuracy being better than ± 10% of the content determined. Examination of the Table shows one or two anomalous results; for instance, sample B shows a discrepancy between the average spectrographic result and the certificated value for the silicon content. No real explanation can be offered for this at the moment, although one suggestion has been put forward to the effect that the sample may contain insoluble silica. This point has to be investigated further. The aluminium figures on sample D are also inconsistent, but here, chemical checks in the authors' laboratory would indicate a value of 0·045% for this sample, which is in much better agreement than the certificated value.

Discussion

This paper has described two methods for the analysis of nickel alloys spectrographically. The first one, due to E. K. Jaycox, has been found to be satisfactory, especially in instances where small sample weights are submitted for analysis. When used on a routine scale, a criticism of the procedure was made on the grounds of the lengthy sample preparation stage required. For this reason, the development of the authors' cup procedure became necessary in order to cut this time requirement to a minimum.

It must not be thought, however, that this is the only justification for the use of the new procedure, there are several other advantages of this direct solution technique. Thus, in routine practice, apart from the ease of sample preparation and of making synthetic standards, there is a marked reduction in the incidence of accidental contamination when low levels of impurities are being determined, as compared with powder techniques. Again, because of the versatility of the cup electrode regarding its use with different types of electrical discharges, there is a possibility that elements present in materials as major constituents, may be analysed for by means of spark sources, with a high order of accuracy. This means that, for example, a steel could be analysed spectrographically in solution, not only for residual impurities, but for alloying constituents as well, simply by suitable choice of discharge conditions and adjustment of solution strength. No reference to chemical analysis is, of course, needed. Thus, the solution procedure reported here, has a wide applicability for spectrographic analysis, and the analysis of nickel alloys is just one example of the possibilities of the procedure.

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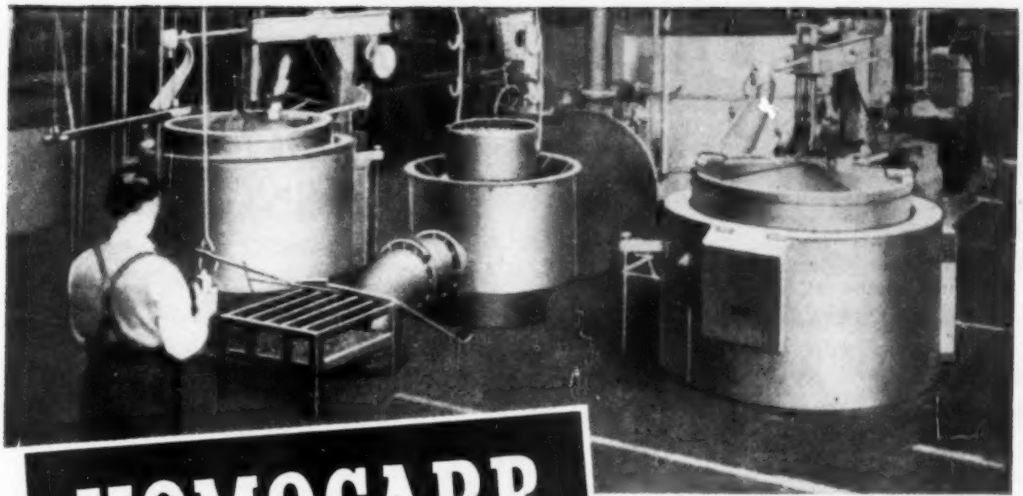

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With a Foreword by E. J. Vaughan, M.Sc., A.R.C.S., D.I.C., F.I.M., F.R.I.C., Director of Materials Research, Admiralty.

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